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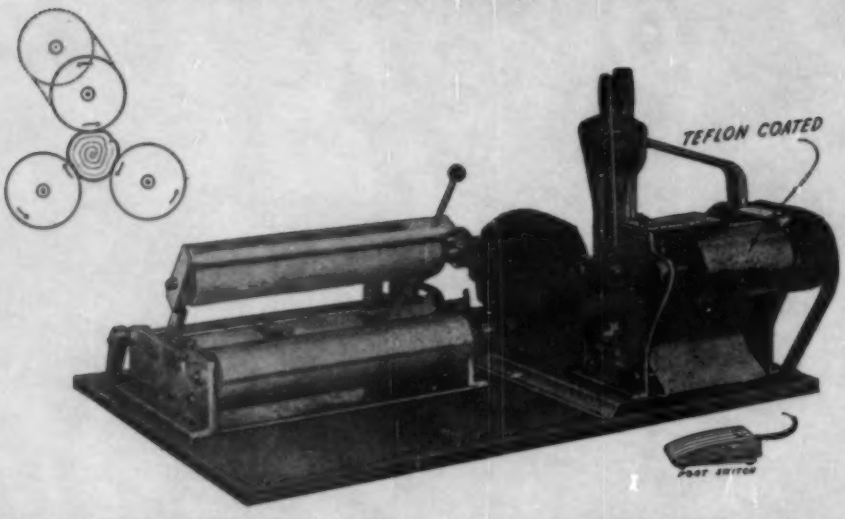

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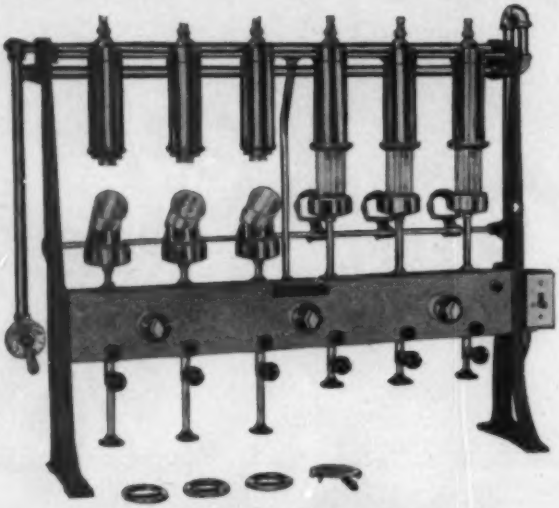

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TO

WILLIAM FINDLAY GEDDES

1896-1961

William Findlay Geddes

WILLIAM FINDLAY GEDDES

Dr. William F. Geddes, Chief of the Department of Agricultural Biochemistry, University of Minnesota, St. Paul, and Editor of CEREAL CHEMISTRY for the past 18 years, died January 7 in Mexico City while on vacation.

As one of the world's leading cereal chemists, Dr. Geddes had been the recipient of numerous honors. In 1958, the Institute of Food Technologists gave him the Nicholas Appert Award "for pre-eminence in and outstanding contributions to Food Technology"; in 1950, he was awarded the coveted Thomas Burr Osborne Medal by the American Association of Cereal Chemists for his "distinguished contributions to the field of cereal chemistry." In a poll taken by the Chicago Section of the American Chemical Society in 1947, he was voted one of the world's "Ten Ablest Food Chemists" and in 1936 he received the Coronation Medal of King George VI.

"Bill" Geddes, as his friends knew him, was born in the small community of Manotick, Ontario, Canada. He attended the University of Toronto and obtained his B.S.A. degree in 1918. Although he had risen to an officers' rank in the Officers' Training Corps, his eyesight did not meet army requirements when he tried to enlist during World War I. Consequently, he became a control chemist in a factory manufacturing sulfuric acid, smokeless powder and T.N.T. It was during this period that he met and became engaged to his wife, Florence May.

After the war, Geddes accepted a position in Winnipeg as Assistant Professor of Agricultural Chemistry at the University of Manitoba. He remained there until 1933 (with two interruptions to obtain an M.A. from the University of Toronto in 1924 and a Ph.D. from the University of Minnesota in 1929) at which time he was Professor and Head of the Department. His first academic interest in cereal chemistry was probably acquired at the University of Minnesota where his Ph.D. thesis concerned the storage of flour and wheat. Thus, it was in keeping with his scientific interests that he accepted a position as chemist-in-charge of the Dominion Grain Research Laboratory in Winnipeg, operated by the Board of Grain Commissioners for Canada.

During the next five years, Geddes made significant contributions to the study of baking tests which were highly effective in producing better methods for the evaluation of wheat flour. This was partially the result of his early work on the bromate effect and its relation to the protein content and quality of Canadian wheats. His statistical studies were also very important in establishing criteria for examining

the results of baking tests. In 1938 he was offered a position as Professor of Agricultural Biochemistry at the University of Minnesota. He accepted this offer and was made Head of the Department in 1944.

In the twenty-three years Dr. Geddes spent at Minnesota, he became a leading authority and researcher on the storage of cereal grains and their products. One of his most outstanding investigations covered the development of the B complex vitamins in the growing and ripening wheat plant. This study was the first of its kind. Probably more important to Dr. Geddes was the time spent in training students, because he was first of all a teacher. The long list of men he taught and trained who have attained influential positions in the fields of industrial management, teaching, and research is alone a memorable achievement.

Next to teaching Geddes enjoyed meeting new people, especially in foreign lands. His wide acquaintances with European cereal chemists began while he was with the Grain Research Laboratory in Winnipeg. On two occasions, he was sent to Europe and each time spent some months in Britain, France, Holland, Germany, and Norway studying their milling and baking practices. In 1952, he and Mrs. Geddes spent an extended vacation on the continent and attended several cereal chemists meetings as a representative of the American Association of Cereal Chemists. Geddes' final trip overseas was made in September of 1960 when he was guest lecturer at the University of Glasgow, Scotland.

During his professional life, Dr. Geddes was sole or joint author of more than 160 scientific papers, 35 technical articles, and author or co-author of chapters in 11 books. He is listed in 10 biographical directories including Who's Who in America. His membership in societies include nine professional and scientific and five honorary and fraternal. In the 32 years he was a member of the AACC he held numerous committee chairmanships and served as President in 1938.

Few men have given to a professional society as much of themselves as did Geddes. His major contribution and perhaps the work he enjoyed most was in the AACC's publications program. He served as Associate Editor of CEREAL CHEMISTRY from 1936 to 1943 and as Editor from 1943 to his death. He established and edited TRANSACTIONS until 1956 when it was discontinued. The AACC's newest publication, CEREAL SCIENCE TODAY, benefited from his advice and guidance during its founding. He was its first editor and served for two years. The Monograph Series of the Association also had Geddes' helping hand either as a committee member or author.

Those of us who worked with him will never forget—so many things.

R. J. TARLETON

CEREAL CHEMISTRY

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THE RELATIONSHIP BETWEEN FARINOGRAPH MOBILITY AND ABSORPTION[1]

J. B. LOUW AND G. N. KRYNAUW

ABSTRACT

Dough mobility bore a parabolic relationship to absorption in farinograph studies of a number of flours when tested over the dough consistency range from 200 to 950 Brabender units. The absorption equivalent to a given interval of farinograph consistency varied for different ranges of consistency and with different flours. The parabolic relationship provides a more satisfactory interpretation of dough behavior in the farinograph than the linear relationship previously reported.

In a previous paper Hlynka (3) presented certain preliminary aspects of dough mobility and absorption, and a linear relationship was reported between dough mobility, obtained as the reciprocal of maximum farinograph consistency, and the absorption, the latter calculated for the two cases of 14% moisture and dry flour basis. The linear relationship was employed in 1) establishing a method for precisely determining the equivalence between consistency and absorption, and 2) evaluating by extrapolation the amount of water in dough that is independent of mobility.

In some preliminary experiments the relation between mobility and absorption appeared to be a parabolic one. This paper reports the results of an investigation of the nature of this relationship using five flours and dough consistencies from 200 to 950 Brabender units (B.u.). A study was also made of the absorption equivalent to a given interval of farinograph consistency over different ranges of consistency for the different flours.

Materials and Methods

The flours used for the investigation were: A, a commercial unbleached flour with an ash content of 0.47%; B, a commercial un-

[1] Manuscript received January 9, 1960. Contribution of The Laboratory, South African Wheat Industry Control Board, Pretoria.

bleached flour with an ash content of 0.81%; C, a commercial bleached flour with an ash content of 0.75%; D, unbleached flour from experimentally milled Manitoba No. 3 wheat with an ash content of 0.56%; and E, an arbitrary blend of unbleached experimentally milled flours with an ash content of 0.65%.

The data were obtained on a farinograph with a large mixing bowl and a 59.0 r.p.m. drive to the bowl. The scale system of the instrument was investigated and proved to be linear.

For each flour sample a series of farinograms were obtained by varying the absorption so that the maximum dough consistencies were obtained in the range 200 to 950 B.u. Consistency readings were taken at the center of the farinograph curve at the point of maximum consistency.

The experiments were conducted according to both the constant flour weight and constant dough weight procedures of the farinograph method for flour as given in *Cereal Laboratory Methods* (1). Flours A and B were carried through both procedures, while only the constant dough weight procedure was used for flours C, D, and E.

The data were calculated in terms of 1) absorption on either a 14% moisture basis or dry matter basis, as indicated; and 2) the dough mobility which was obtained as the reciprocal of the maximum consistency and expressed in reciprocal Brabender units.

The dough mobilities were plotted graphically against the absorption, the latter being regarded as the independent variable.

Results

Constant Flour Weight Procedure. In this case the results have bearing on the practical use of the farinograph, and it was deemed sufficient to calculate the absorption only on a 14% moisture basis. The resulting graphical relationships are given in Fig. 1.

From the curves it was clear that a linear relationship was inade-

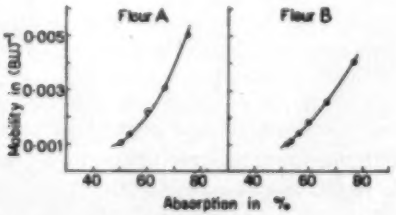

Fig. 1. Variation of dough mobility with absorption on 14% moisture basis using the constant flour weight procedure.

quate for representing the behavior of the curves. Consequently a mean square regression curve was sought to fit the data of each flour. For the relationship between dough mobility and absorption on a 14% moisture basis the following general equation was considered:

$$M = As^2 + Bs + C \quad (1)$$

where M = dough mobility measured by the reciprocal of the maximum consistency of the farinogram and expressed in reciprocal Brabender units;

s = absorption expressed as percent of flour (14.0% moisture basis);

A, B, C = constants.

Assuming that the error lies only in the values of the ordinates M_i, the method of least squares was applied to obtain the best-fitting curve (2). Evaluation of the constants A, B, and C led to the following equations:

$$M = 3.724 \times 10^{-6} s^2 - 3.105 \times 10^{-4} s + 7.340 \times 10^{-3} \quad (2)$$

for flour A, and

$$M = 1.679 \times 10^{-6} s^2 - 9.533 \times 10^{-5} s + 1.496 \times 10^{-3} \quad (2a)$$

for flour B.

These equations gave accurate information for interpolated values in the experimental range of the curves.

The change in absorption corresponding to a change of 20 B.u. in dough consistency was then considered. Using equations (2) and (2a), the absorption for dough consistencies in the range 400 to 600 B.u. was calculated at intervals of 20 B.u. This was done by solving the quadratic equation (1), which by rearrangement gives

$$As^2 + Bs + C - M = 0$$

where M is the dough mobility corresponding to the particular dough consistency. The solution for the absorption is

$$s = \frac{-B + \sqrt{B^2 - 4A(C - M)}}{2A}$$

The results for flours A and B are summarized in Table I.

Constant Dough Weight Procedure. In this case varying absorptions were obtained by using different proportions of flour and water to give a total dough weight of 480 g. The dough mobilities were plotted against the corresponding absorptions calculated on dry-matter basis for both flours A and B as well as the other three flours, C, D, and E. The graphical relationships are given in Fig. 2, and it was clear that they were of the same form as those in Fig. 1.

TABLE I

THE ABSORPTION EQUIVALENT TO 20 BRABENDER UNITS OF FARINOGRAPH DOUGH CONSISTENCY IN THE RANGE 400 TO 600 BRABENDER UNITS

Consistency	Flour A		Flour B	
	Absorption, 14% Moisture Basis	Absorption Equivalent to 20 B.u.	Absorption, 14% Moisture Basis	Absorption Equivalent to 20 B.u.
B.u.				
400	62.64		65.87	
		0.78		0.96
420	61.86		64.91	
		0.73		0.89
440	61.13		64.02	
		0.70		0.84
460	60.43		63.18	
		0.66		0.78
480	59.77		62.40	
		0.63		0.74
500	59.14		61.66	
		0.60		0.69
520	58.54		60.97	
		0.58		0.66
540	57.96		60.31	
		0.55		0.62
560	57.41		59.69	
		0.54		0.60
580	56.87		59.09	
		0.51		0.56
600	56.36		58.53	

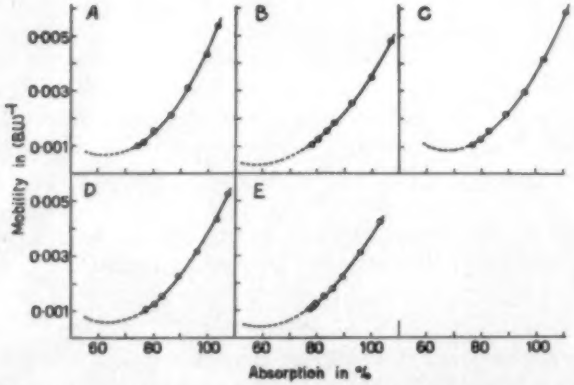

Fig. 2. Variation of dough mobility with absorption on dry-matter basis using the constant dough weight procedure. The broken portions of each curve represent extrapolation of the curves as computed from the regression equations for the experimental data for mobility and dough absorption.

Utilizing the general equation (1) and substituting for s the absorption which gives the total water content of the dough as a percentage

of the dry matter, the following regression curves were obtained:

$$\text{Flour A: } M = 2.719 \times 10^{-6}\ s^2 - 3.386 \times 10^{-4}\ s + 1.119 \times 10^{-2} \quad (4)$$
$$\text{Flour B: } M = 1.831 \times 10^{-6}\ s^2 - 2.109 \times 10^{-4}\ s + 6.397 \times 10^{-3} \quad (4a)$$
$$\text{Flour C: } M = 2.636 \times 10^{-6}\ s^2 - 3.547 \times 10^{-4}\ s + 1.280 \times 10^{-2} \quad (4b)$$
$$\text{Flour D: } M = 2.431 \times 10^{-6}\ s^2 - 3.083 \times 10^{-4}\ s + 1.036 \times 10^{-2} \quad (4c)$$
$$\text{Flour E: } M = 1.980 \times 10^{-6}\ s^2 - 2.342 \times 10^{-4}\ s + 7.336 \times 10^{-3} \quad (4d)$$

The curves were extrapolated beyond their turning points by making use of the above equations. The extrapolations are shown by the broken lines in Fig. 2, where the parabolic nature of the dough mobility-absorption relationship becomes clear.

The absorption at the minimum of the curve is given by

$$s = -\frac{B}{2A},$$

and by using this expression, the absorptions corresponding to the extrapolated minimum mobilities were evaluated. The results are given in Table II.

TABLE II

ABSORPTION CORRESPONDING TO EXTRAPOLATED MINIMUM MOBILITY

FLOUR	ABSORPTION DRY-MATTER BASIS
	%
A	62.25
B	57.57
C	67.29
D	63.43
E	59.13

Discussion

In view of the experimental results obtained in the present investigation, a modified presentation of the findings by Hlynka (3) becomes necessary. The latter's findings were based on experimental results obtained in the range 300 to 900 B.u. of the farinograph consistency scale. Even in that range the authors found a definite curvature trend which was emphasized by extending observations from 200 to 950 B.u. It therefore appears that dough mobility is not a linear function of absorption. Furthermore, the result of a linear relationship is that extrapolation leads to an intercept on the absorption axis where the dough mobility is zero. The implication is that consistencies of infinite value can be obtained, whereas such situations are never realized within the system under consideration.

The parabolic relationship, although of limited quantitative ap-

plicability when extrapolated to low absorption values, seems to give a rendering of the correct form for the behavior of the system. On decreasing the absorption in practice, a maximum dough consistency (minimum dough mobility) is reached beyond which homogeneous mixing of the flour and water is not attainable, but in the extreme case of dry flour the consistency decreases to a very low value.

A significant feature of the parabolic relationship is that a minimum mobility is characteristic of the relationship. Furthermore, this minimum can be interpreted in terms of the explanation advanced by Hlynka for the intercept value obtained by the linear relationship. The absorption value at the minimum dough mobility may be regarded as the quantity of water absorbed by the starch granules and bound by the hydration of the proteins. Qualitatively these processes would be expected to decrease the dough mobility. After maximum absorption and hydration have been obtained, the further addition of water should increase the mobility, as is also shown by the parabolic relationship.

It must be pointed out, however, that the structure of the flour-water system changes during the hydration process and is further modified by the mechanical treatment. This can be responsible for the breakdown of the quantitative rendering of the behavior of the system by the parabolic relationship at low absorption values, for, when merely 480 g. of flour with approximately 14% moisture content were mixed in the mixing bowl, a consistency of about 30 B.u., corresponding to a dough mobility of 33.3×10^{-3} reciprocal B.u., was obtained irrespective of the flour. It therefore appears that this change in structure opens to criticism any continuity sought between the behavior in the farinograph of a flour-water system at a low water content and its behavior at a high water content. It is possible to trace the discontinuity to the point where the water content of the system is insufficient to form a homogeneous dough. The parabolic relationship deduced from a system with a dough structure should therefore be expected to be valid for flour-water systems only if the systems consist of such a structure. It was found for the flours with low absorption at their minimum mobilities, namely flours B and E, that a very stiff though homogeneous dough structure could only just be obtained by manual manipulation of a dough with a water content corresponding to the respective absorption at minimum mobility. The flours with higher absorption values at minimum mobility could be more readily worked into doughs of which the water content corresponded to their absorption values at minimum mobility. In this connection it is perhaps of significance that a comparison of

the curves in Fig. 2 shows that the doughs with higher absorption values at their minimum mobilities had higher minimum mobilities than the doughs with lower absorption values. The parabolic relationship would therefore appear to be applicable, quantitatively at least, up to the turning point of its extrapolation.

According to the results in Table II the absorption value at a minimum dough mobility varies from flour to flour with an average value in the region of 62%. This figure is evidently dependent upon protein content and quality, starch behavior, and rate of extraction. Thus it is improbable that a constant figure is obtainable even for flours of the same rate of extraction. The average value of 62% differs by 7% from the single value of 69% reported by Hlynka (3). The value obtained by the linear relationship, however, depends upon the range of consistencies used which determines the slope and thus the intercept of the regression line.

This investigation also shows that the absorption which is equivalent to a given change in dough consistency depends upon the particular flour. This is borne out in the figures given in Table I. This quantity, however, does show the general trend of decreasing in going from low to high consistencies for any given flour, but the dependence on the flour used excludes the deduction of identical values for different flours for this trend.

The parabolic relationship between dough mobility and absorption gives a more accurate expression of dough behavior than a linear relationship, in that it lends itself to a more accurate rendering of the dough behavior in the experimental portion of the curve and to a more feasible interpretation of its extrapolated form.

Further investigations necessary to relate the relationship to more fundamental concepts are in progress.

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MEASUREMENT OF FLOUR WHITENESS[1]

A. W. CROES[2]

ABSTRACT

The whiteness of wheat flour was determined by means of photoelectric tristimulus measurements and substitution of the reflectance values A, B, and G in a newly derived equation for whiteness. This formula, which is in its most simple form $W = G - A + B$, simplifies the calculation of whiteness considerably, and, therefore, should be considered as useful for routine application. The results obtained were in complete accord with the visually estimated orders as found by means of the Pekar test. They can be considered as representative for visual judgments in average daylight.

The whiteness of flour is governed by two independent factors, namely lightness ("brightness") and degree of yellowness (saturation or departure from neutral white).

The lightness is related to the amount of bran content that is present in the flour, but it is also influenced by the granularity (particle size) of the flour. The degree of yellowness is dependent upon the amount of carotene and xanthophyll in the original wheat and the extent to which the flour has undergone natural and artificial bleaching.

The earliest and most widely used method of assessing the color of flour is the Pekar test. Although this visual test undoubtedly is very useful, it does not permit fixation of the results in objective figures. On account of this drawback, attempts have been made to devise methods of evaluating the color of flour and to record the results on a numerical basis. Concerning whiteness most of these methods, however, are limited to the determination of one of the two factors governing whiteness.

The Kent-Jones & Martin Flour Color Grader is a reflectometer, especially designed for the milling industry. It enables the miller to be informed quickly about the proportion of branny matter in the flour, since "color grade" is approximately correlated with degree of extraction. To eliminate the influence of granularity, the flour is made into a smooth paste by mixing together 30 g. of flour and 50 ml. of distilled water. Immediately after mixing, this paste is poured into a glass cell and the amount of light reflected, after passing a green filter, is recorded by means of a photoelectric cell and galvanometer. The

[1] Manuscript received June 30, 1959.
[2] Institute for Cereals, Flour, and Bread T.N.O., Wageningen, The Netherlands.

color grade of the flour is then read off on a scale (4,5,6).

The green filter of the Kent-Jones & Martin Flour Color Grader has its peak transmission at 530 mμ. As maximum visibility for a normal human eye occurs at 555 mμ, this instrument measures the approximate lightness. The lightness is that attribute of a color which permits it to be classed as equivalent to some member of the series of grays ranging from absolute black (zero) to pure white (100). In expressing the visual impression of whiteness, however, the lightness alone is inadequate, since the degree of yellowness should also be taken into account.

Materials and Methods

With a photoelectric tristimulus reflectometer (2) it is possible to determine lightness and degree of yellowness. This instrument is based on the principles of the CIE[3] standard system for designating colors. On the premise that the visual judgments are made in average daylight, it duplicates an observer, in fact the CIE standard observer, by a source-photocell combination with three filters. The only difference in operating procedure from the Kent-Jones & Martin Flour Color Grader is that on each sample three readings instead of one have to be taken. These readings are the reflectance values A, B, and G, obtained by measuring with the amber, blue, and green filter of the tristimulus set. The setting with the tristimulus green filter is identical with lightness in the CIE meaning. The degree of yellowness can be computed from the equation for yellowness as given by Hunter (2): Yellowness = (A − B)/G.

In general, the whiteness increases if the lightness increases or if the degree of yellowness decreases. But, in expressing the visual impression of whiteness, it is necessary to have one figure in which both the positive effect of lightness and the negative effect of yellowness are taken into account. The calculations of whiteness with the formulas mentioned in literature, however, are rather complicated and are, therefore, to be considered as less suitable for use in practice.

Whiteness can be calculated directly and in a simple way by using formula

$$W = G - A + B, \qquad (W')$$

in which A, B, and G are the three tristimulus reflectance values. This formula was derived theoretically from the CIE standard system, taking into account the mentioned equation for yellowness (1).

Formula $W = G - A + B$ was tested in the following way. With a tristimulus reflectometer the reflectance values A, B, and G were

[3] Commission Internationale de l'Eclairage.

determined with a series of six samples of wheat flour of different origin (Table I), six samples of low-grade flour (wheat feed) of different origin (Table II), one sample of flour, bleached with five different quantities of benzoyl peroxide (Table III), and one sample of flour, bleached with five different quantities of chlorine dioxide (Table IV). Whiteness was calculated from the newly derived formula, and also from the equation recommended by Hunter (2) for the calculation of whiteness from data obtained with the tristimulus reflectometer:

$$W = 1 - \left[\left\{ 8.6 \ (a^2 + \beta^2)^{1/2} \right\}^2 + \left\{ \frac{(1.00 - Y)}{2} \right\}^2 \right]^{1/2}, \qquad (W'')$$

in which $a = 2.5 \ (A - G) : 10$ G, $\beta = (G - B) : 10$ G and $Y = G$.

This equation was derived by Hunter from the formula suggested by Judd (3):

$$W = 1 - \frac{1}{K} \Delta E_{\text{MgO to specimen}}$$

Originally K was given the value 20 and in Hunter's formula this value was used. Considering Judd's remark (3), however, that the value of K should be set much higher than 20 if the visual judgments are made on samples with a fine dividing line, the value 70 was chosen as proposed by Selling and Friele (7). In that case the numerical coefficient in Hunter's formula becomes 8.6 instead of 30.

The orders of whiteness were also determined visually by seven persons with the familiar Pekar test for milling products. As the measurements with the tristimulus reflectometer were made on pastes, obtained by stirring 30 g. of flour with 50 ml. of distilled water, the visual estimation was brought in line with them by determining the orders of whiteness on moistened material.

Results and Discussion

In Tables I, II, III, and IV the measured reflectance values (Amber, Green, Blue) are given, as well as the figures for Lightness, Yellowness, and Whiteness (W′, W″) computed from them. For convenience the results obtained by means of Hunter's formula for whiteness (W″) are expressed as a percentage. The orders of lightness, yellowness and whiteness are indicated by means of the figures 1 to 6: 1 = highest degree of lightness, lowest degree of yellowness, or highest degree of whiteness; and 6 = lowest degree of lightness, highest degree of yellowness, or lowest degree of whiteness. For comparison the results of the visual assessments are given in the last column

TABLE I

RESULTS OF TRISTIMULUS MEASUREMENTS ON SIX SAMPLES OF WHEAT FLOUR OF DIFFERENT ORIGIN
(Numbers in parentheses indicate order or sequence)

No.	Material	Reflectance Values[a]			Lightness[b]	Yellowness[c]	W'[d]	W''[d]	Pekar[e]
		Amber	Green	Blue					
1	Patent flour	65.4	64.3	56.8	64.3(2)	0.134(1)	55.7(1)	79.2(1)	1
2	Patent flour	65.7	64.4	56.7	64.4(1)	0.140(2)	55.4(2)	79.0(2)	2
3	Imported flour	65.1	63.6	55.2	63.6(3)	0.156(3)	53.7(3)	78.0(3)	3
4	Imported flour	64.4	62.9	54.5	62.9(4)	0.157(4)	53.0(4)	77.6(4)	4
5	Baker's grade flour	63.8	62.4	53.4	62.4(6)	0.167(5)	52.0(5)	77.0(5)	5
6	Baker's grade flour	63.9	62.5	53.1	62.5(5)	0.173(6)	51.7(6)	76.7(6)	6

[a] Amber, Green, and Blue: respectively, reflectance value with the tristimulus amber, green, and blue filters.
[b] Lightness: G.
[c] Yellowness: (A − B)/G.
[d] Whiteness: $W' = G - A + B$. $W'' = 1 - [(8.6(a^2 + \beta^2)^{1/2})^2 + ((1.00 - Y)/2)^2]^{1/2}$.
[e] Visual assessment of whiteness by means of the Pekar test on moistened material.

TABLE II

RESULTS OF TRISTIMULUS MEASUREMENTS ON SIX SAMPLES OF LOW-GRADE FLOUR (WHEAT FEED) OF DIFFERENT ORIGIN[a]
(Numbers in parentheses indicate order or sequence)

No.	Material	Reflectance Values			Lightness	Yellowness	W'	W''	Pekar
		Amber	Green	Blue					
1	Low-grade flour	52.1	49.5	35.8	49.5(1)	0.329(1)	33.2(1)	63.5(1)	1
2		50.5	47.9	34.7	47.9(2)	0.330(2)	32.1(2)	62.9(2)	2
3		46.4	43.7	30.7	43.7(4)	0.359(3)	28.0(3)	59.7(3)	3
4		46.1	43.5	30.0	43.5(5)	0.370(5)	27.4(4)	59.1(4)	4
5		45.4	42.5	30.0	42.5(6)	0.362(4)	27.1(5)	59.0(5)	5–6
6		47.7	44.1	30.6	44.1(3)	0.388(6)	27.0(6)	57.8(6)	5–6

[a] See footnotes, Table I.

TABLE III

RESULTS OF TRISTIMULUS MEASUREMENTS ON A SAMPLE OF FLOUR BLEACHED WITH FIVE DIFFERENT QUANTITIES OF BENZOYL PEROXIDE[a]
(Numbers in parentheses indicate order or sequence)

No.	Amount of Bleaching Agent	Reflectance Values			Lightness	Yellowness	W'	W''	Pekar
		Amber	Green	Blue					
	g/100 kg								
1	Blank	59.5	58.1	46.6	58.1(5)	0.222(6)	45.2(6)	72.5(6)	6
2	0.75	59.3	58.0	47.0	58.0(6)	0.212(5)	45.7(5)	73.0(5)	5
3	1.50	59.8	58.3	48.4	58.3(4)	0.196(4)	46.9(4)	73.9(4)	4
4	2.25	59.7	58.5	48.5	58.5(1–2)	0.192(3)	47.3(3)	74.2(3)	3
5	3.00	59.4	58.4	49.1	58.4(3)	0.176(2)	48.1(2)	74.8(2)	2
6	3.75	59.7	58.5	49.7	58.5(1–2)	0.171(1)	48.5(1)	75.2(1)	1

[a] See footnotes, Table I.

TABLE IV

RESULTS OF TRISTIMULUS MEASUREMENTS ON A SAMPLE OF FLOUR BLEACHED WITH FIVE DIFFERENT QUANTITIES OF CHLORINE DIOXIDE[a]
(Numbers in parentheses indicate order or sequence)

No.	Amount of Bleaching Agent	Reflectance Values			Lightness	Yellowness	W'	W''	Pekar
		Amber	Green	Blue					
	mg/kg								
1	Blank	62.3	60.7	49.3	60.7(3)	0.214(6)	47.7(6)	73.9(6)	6
2	10	62.2	60.6	50.7	60.6(4)	0.190(5)	49.1(5)	75.2(5)	5
3	20	62.2	61.0	52.1	61.0(1–2)	0.166(4)	50.9(3)	76.4(4)	3
4	30	62.4	61.0	52.9	61.0(1–2)	0.156(3)	51.5(1)	76.9(1)	1
5	50	62.0	60.4	53.0	60.4(5)	0.149(2)	51.4(2)	76.8(2)	2
6	100	60.6	59.2	52.0	59.2(6)	0.145(1)	50.6(4)	76.5(3)	4

[a] See footnotes, Table I.

(Pekar): 1 = highest degree of whiteness, and 6 = lowest degree of whiteness.

The orders of whiteness, resulting from the calculations with both formulas, were in agreement with the visual assessments by means of the Pekar test, apart from one change of place of two samples in Table IV where Hunter's formula was used.

The orders of lightness are in general very different from the visual arrangements according to Pekar. In Table IV there is a noticeable decrease in lightness in samples 5 and 6, owing to an overdose of chlorine dioxide and also effecting a decrease in whiteness.

The orders of yellowness are in considerably better agreement with the results of the Pekar test, but on the whole the results confirm the fact that, in evaluating a figure for whiteness, both lightness and yellowness must be taken into account.

In Tables III and IV, the effect of bleaching agents can be followed from the calculated figures for yellowness.

The use of formula $W = G - A + B$ means a considerable simplification of the calculation of whiteness and facilitates practical application of the tristimulus method for objective whiteness measurements. The results obtained were in complete accord with the visually estimated orders as found by means of the Pekar test. They can be considered as representative for visual judgments in average daylight.

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AN APPARATUS FOR MEASURING THE TORSIONAL STRENGTH OF MACARONI[1]

L. P. KARACSONYI AND A. C. BORSOS[2]

ABSTRACT

A device is described for measuring the torsional strength of macaroni and spaghetti. The standard error of the means of ten measurements was 1.79 kg. per cm.^{2} A highly significant correlation of + 0.85 was found between torsional strength and bending strength of 25 samples of macaroni, but neither of these properties was significantly correlated with the "wet weight" of the macaroni after cooking. However, the smaller the value of the torsional or the bending strength, the more observable were such defects as inelasticity, splitting, checking, or tendency to become sticky upon cooking.

This device is particularly useful for detection of hidden failures in macaroni. In a macaroni specimen a fissure running in a straight line along the axis of the cylinder, or very fine irregular cracks or checks, can exist. Such a macaroni may show a rather high bending strength, but when twisted, just as during cooking, it disintegrates.

The quality of a macaroni sample is usually ascertained by determining its cooking and mechanical characteristics. However, the majority of the methods currently in use are subjective. Such cooking characteristics as color, smell, and taste can only be evaluated by organoleptical means. The elasticity and ease of mastication of the cooked product are ascertained by subjective test. The only mechanical test which has been studied extensively is the bending or breaking strength (1–6).

A routine test known for a long time is to twist a macaroni specimen with both hands in opposite directions. This otherwise simple and practical method is deficient in two respects: 1) it is subjective, so that the result of the test depends to a large extent on expert and skillful handling by the operator; and 2) the result cannot be expressed either by a number or a diagram.

The present study was undertaken to design and construct an apparatus, as simple as possible, which would ensure that the twisting can always be performed under identical conditions and that the test results can be expressed by a single number.

The torsional strengths of 25 samples of macaroni were determined and the data compared with the bending strength and cooking characteristics of the samples.

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Description and Use of the Torsionmeter

The main features of the torsionmeter are shown in Figs. 1 and 2. It consists of a phonograph motor *1* with a coupled reducing gear connected through a clutch *2* to a clamping head which is rotated at 3 r.p.m. After the specimen has been clamped, the clutch, *2*, is displaced to the left and the motor rotates the sleeve *4* placed on shaft *3*. The torque is transmitted by the crosspin *5*. A longitudinal slot is provided in sleeve *4* so that the pin may have free axial movement. This is necessary for clamping the specimen and also for balancing axial stresses. The two clamping heads are identical and are illustrated in Figs. 2 and 3. Two small pins join the forked piece *6* to sleeve *4* (Figs. 1 and 2). Two tapered pins *7* which are adjustable by means of screws, retain the clamping head *8* in fork *6*; in this way, the clamping heads *8* may adjust themselves freely in a similar manner to ball joints. Sleeve *4* of the clamping head (Figs. 1 and 2) is short in length and is keyed to shaft *9* (Fig. 3), which supports the loading arm *10* (Figs. 1 and 2) producing the torque. The loading weight is about 150 g. and in Fig. 2 it is shown suspended at the shorter arm length used for testing specimens of very thin alimentary pastes.

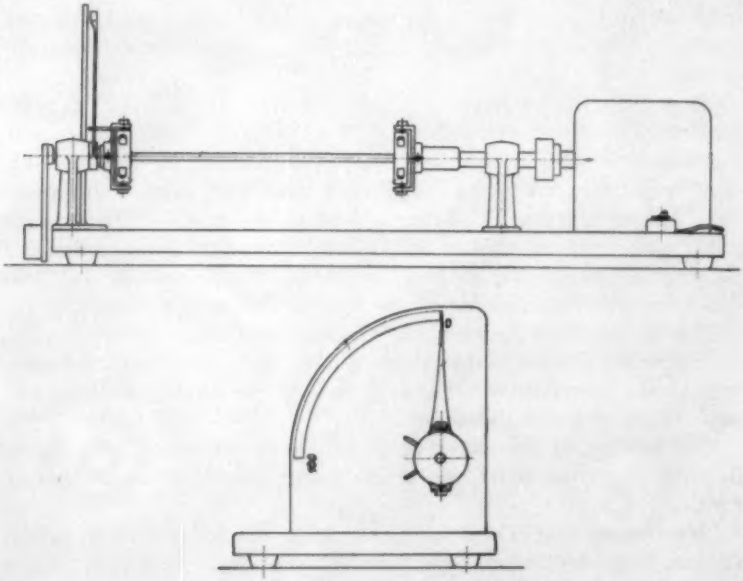

Fig. 1. Diagram of torsionmeter.

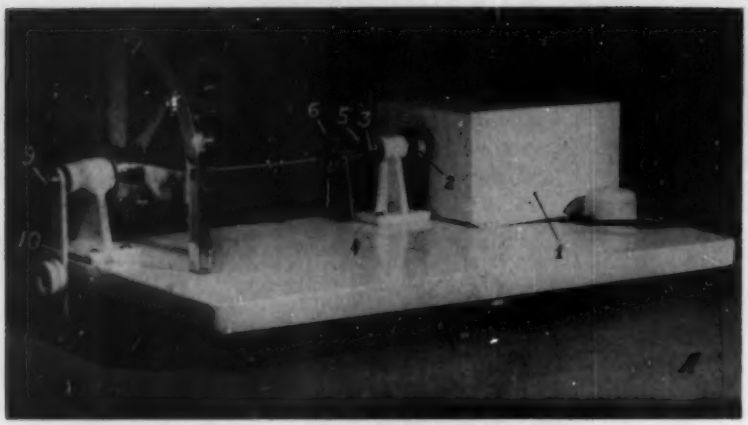

Fig. 2. Torsionmeter.

With thick samples of high strength, the weight should be placed at the greatest arm length; in some cases arm extensions should be used. As shown in Fig. 3 the index pointer *11* is dragged along by pin *12* by means of the small plate, *13*, mounted to the index. The index pointer can only be displaced by pin *12* in one direction. The balanced index pointer moves very easily and remains in the position attained until the sample is broken by the torsion to which it has been subjected.

It is advisable to make an auxiliary calibrating disk for scale recording. This is a light disk of some 150-mm. diameter, built onto the loading arm. A flexible thread is run on the disk perimeter, fixing one end to the disk and suspending a light load pan on the other. Progressively increased loads are placed in the pan and the relevant index position is recorded on the scale. The product of disk radius and loading weight specifies loading torque in units of cm. per kg. Two scales are plotted according to the two possible load positions.

Three jaws grip the sample. The clamping effect is self-locking, i.e., increased friction of the clamping jaws – as a consequence of specimen twist – increases the clamping force of the springs. Knurled jaw surfaces will enhance this action.

The bearing on the scale side should be of extremely low friction in order to minimize friction effects which would influence the test results.

Measurement of Torsional Strength. The specimen of macaroni (20 cm. long) is clamped between the two clamping heads in such a way that the heads can rotate freely in either direction. The pointer

Fig. 3. Clamping head and index.

is set to the zero mark of the dial plate, and the motor is started and allowed to run until the macaroni breaks. After having broken the specimen, the pendulum returns to the vertical position, and the pointer remains in the position reached at the moment of the break. The measurement is repeated ten times with different specimens of the macaroni, and the mean torsional strength in kg. per cm.^{2} is calculated from the following formula:

$$\tau = \frac{D}{D^4 - d^4} \cdot \frac{16\, M_t}{\pi} \sin \alpha$$

where D = the outer diameter of the macaroni, in cm.;
d = the inner diameter of the macaroni, in cm.;
M_t = the product of the weight of the loading pendulum by the

length of the moment arm, in cm. per kg.;
α = angular deflection of the pointer.

The scale is calibrated in terms of $M_t \sin \alpha$; thus the scale values multipled by $(16/\pi) = 5{,}093$ give the actual torque in cm. per kg. The torsional strength of macaroni is equal to the product of the quotient $D/(D^4 - d^4)$ by 5,093 and the number read on the scale.

With the clamping heads provided, specimens of macaroni or spaghetti having a diameter of 2 to 7 mm. can be fixed. In testing spaghetti the loading pendulum should be set, by means of an adjusting knob, to the position of low torque, and the scale showing the smaller values should be used for test evaluation. In this case the quotient is $1/D^3$ instead of $D/(D^4 - d^4)$.

Materials and Methods

Materials. All macaroni samples under investigation were made from Ogg flours of more or less hard aestivum wheats, with an average particle size of 240 μ and an ash content between 0.50 and 0.55%. With the exception of No. 21, most macaroni samples were manufactured on a commercial scale by using two eggs per kg. of flour. Particulars regarding other individual samples are given in Table I.

Methods: Bending strength. The bending strength was determined according to the principle of Lukianov (3) on a Gruzl laborograph modified for this purpose by Szalai (6).

Lukianov places the macaroni specimen on a channel-shaped holder fixed to a pedestal, with supporting points 150 mm. apart. A pan of known weight is suspended on the specimen, against an index along the center line of the specimen holder. Weights of 10 g. each are placed on the pan until the specimen breaks. A measure of bending strength is given by the sum of the pan weight and the maximum break-free weights added to the pan. The test is repeated ten times and the arithmetic mean computed.

In the modification proposed by Szalai (6), the specimen is broken by the motor of the Gruzl-system laborograph. The height of the diagram recorded by the apparatus gives specimen break load in g.

The formula follows:

$$\sigma = \frac{D}{(D^4 - d^4)} \cdot \frac{8\,1\,P}{\pi}$$

or in a simplified form,

$$\sigma = \frac{D}{(D^4 - d^4)}\,39\,P$$

TABLE I

MECHANICAL AND COOKING CHARACTERISTICS OF MACARONI SAMPLES

Number[a] and Type	Torsional Strength	Bending Strength	Cooking Time	Wet Weight	Remarks
	kg/cm²	kg/cm²	minutes	g.	
1 Partly gelatinized flour	73.3	247	11.0	26.1	Slightly sticky
2 Liquid yeast	63.9	290	11.5	25.4	
3 Partly gelatinized flour	56.2	263	10.5	29.1	10% split in two, sticky
4 Straight-grade flour	52.8	228	9.5	24.7	Slightly sticky
5 Contains eggs	46.3	175	8.0	28.6	
6 75% Farina +25% flour	43.3	146	12.0	29.5	Sticky
7 Contains eggs	35.2	129	10.5	28.2	
8 Contains eggs	33.9	147	10.5	24.7	A few specimens split in two
9 Contains eggs	31.7	139	9.5	24.7	
10 Contains eggs	28.9	156	11.0	22.0	Slightly sticky
11 Contains eggs	25.0	144	12.0	22.5	Sticky. A few specimens split in two
12 Contains eggs	23.6	129	18.0	27.2	
13 Contains eggs	23.4	124	18.0	27.4	
14 Contains eggs	23.2	138	20.0	27.8	
15 Contains eggs	21.9	151	21.0	28.2	
16 Contains eggs	20.5	155	20.0	27.9	
17 Contains eggs	19.9	136	19.0	27.6	
18 Contains eggs	19.4	108	10.0	21.8	Sticky
19 Contains eggs	16.0	144	18.0	25.7	A few specimens split in two
20 Contains eggs	15.8	142	17.0	26.4	Pulpy
21 Plain	14.8	143	15.0	24.4	Checked, inelastic
22 Contains eggs	14.6	151	18.0	25.1	Inelastic
23 Contains eggs	12.7	157	21.0	26.6	Somewhat pulpy
24 Contains eggs	10.8	120	20.0	27.0	Checked, inelastic
25 Contains eggs	8.8	134	16.0	22.3	Pulpy, inelastic

[a] Samples 1 through 7, 20, 21, and 25: o.d. 0.51 cm.; i.d. 0.29 cm. Samples 8 through 19, 22, 23, and 24: o.d. 0.60 cm.; i.d. 0.25 cm.

where D = the outer diameter of the macaroni in cm.;
d = the inner diameter of the macaroni in cm.;
l = the distance of the supporting edges in cm. (here 15 cm.);
P = the load necessary to break the macaroni specimen in kg.

Cooking Time and Cooking Characteristics. For determining the cooking time and cooking characteristics 10 g. of macaroni are cooked

in 100 ml. of water to an equal grade of tenderness, filtered on a Büchner funnel, and rinsed with lukewarm water two or three times. After filtration and cooling the macaroni is weighed and examined for possible discontinuity, appearance of the surface and cross section, elasticity, and ease of mastication.

The equal grade of tenderness is determined by gently pressing a macaroni or spaghetti specimen under cooking against the side wall of the beaker with a glass rod. Cooking is complete when the specimen can be cut fairly easily in this manner.

Results and Discussion

The mechanical and cooking characteristics of 25 samples of macaroni representing several types are recorded in Table I. The standard error of the mean values for torsional strength, computed from the variance for "within samples," was 1.8 kg. per cm^2. This error is influenced not only by the reproducibility of the measuring technique but also by the lack of uniformity in the test specimens. A bran particle or an air bubble will cause wide variations in mechanical strength and it is necessary to make several measurements on each lot of macaroni or spaghetti to obtain meaningful values.

Simple correlations computed between the two indices of mechanical strength and wet weights of the cooked samples are given below:

Nature of Correlation	r^a
Torsional strength × bending strength	+0.85
Torsional strength × wet weight	+0.22
Bending strength × wet weight	+0.12

[a] Value of r at 5% point = 0.38; at 1% point = 0.49.

The correlation between the two measures of mechanical strength is highly significant, but these data are not associated with the wet weight of the samples. The latter finding is rather surprising, since the quality of macaroni products is usually based upon the determination of the swelling power, that is, the increase in weight (wet weight) upon cooking. However, the data in Table I show a rather remarkable parallelism between torsional and bending strength along with diverse cooking characteristics. Nevertheless, samples of low mechanical strength invariably have undesirable properties such as checking or splitting of the uncooked macaroni and an inelastic, pulpy product upon cooking.

For evaluating the mechanical characteristics of macaroni, the assessment of torsional strength and bending strength appears to be equally useful. There are cases, however, in which the bending strength value does not clearly indicate the real quality of the

product. For example, a fissure can occur in a macaroni specimen which runs in a straight line along the axis of the cylinder; also, there may be fine irregular cracks, called checks, that can hardly be seen by the naked eye. Such a macaroni could show a rather high bending value but a low twisting strength; during cooking, it would doubtless disintegrate. This opinion is supported by data obtained in storage trials. In the case of one sample, the bending strength decreased only 27% (from 223 to 162 kg. per cm.^{2}) during about 4 months' storage; its torsional strength, however, decreased 65% (from 25.7 to 9.1 kg. per cm.^{2}). In another sample the initial and final bending strength values were the same (135 and 134 kg. per cm.^{2} respectively), whereas the torsional strength showed a reduction of 66.5% (from 26.2 to 8.8 kg. per cm.^{2}) during the same interval. The behavior of these products during cooking supported the evaluation obtained by measuring the torsional strength. It may be concluded that the measurement of torsional strength gives an accurate index of the quality of macaroni samples, even in those cases where the determination of bending strength does not yield a reliable value.

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STEEPING STUDIES WITH CORN ENDOSPERM SECTIONS[1]

S. A. Watson and E. H. Sanders

ABSTRACT

A technique is presented for studying the effect of variables in the steeping medium on the release of starch granules from corn endosperm. Corn kernels are softened in a water-saturated atmosphere and 10-micron sections are cut from the horny endosperm. The sections are suspended with gentle agitation in dilute solutions of potassium metabisulfite at pH 4 and 52° or 60°C. Sections are removed at intervals during a 6-hour steeping period and stained with iodine. Extent of starch release is measured photometrically and plotted against steeping time. This technique measures directly the tenacity with which the protein matrix holds the starch granules.

In the absence of sulfur dioxide, no starch was released from the sections. Maximum rate and extent of starch release were obtained from corn samples steeped in solutions containing 0.15 to 0.4% sulfur dioxide. Corn dried from 32% to 12% moisture in air at 120°F. (49°C.) gave normal starch release, but corn dried at 200°F. (93°C.) gave very little starch release under any steeping conditions. Corn dried at 180°F. (82°C.) and steeped at 52°C. (optimum commercial steeping temperature) with 0.2% sulfur dioxide, released two-thirds as much starch as the corn dried at 120°F. (49°C.).

In the manufacture of corn starch, the corn is first softened by steeping for 40–48 hours at about 50°C. in water containing 0.1 to 0.2% sulfur dioxide. The corn is then ground and processed for separation of starch, gluten, germ, and hulls. It is sometimes desirable to determine, in the laboratory, steeping conditions that will give optimum starch recovery for a given lot or type of corn. This is usually done by simulating commercial steeping and milling in order to determine the yield and quality of starch and other fractions (2,4,8). Since the steeping conditions affect specific component structures of cells and tissues, microscopic evaluation of sections of corn kernels should give the desired information directly.

Cox, MacMasters, and Hilbert (1) cut microtome sections of steeped corn kernels and microscopically examined the condition of the endosperm. They found that the extent of swelling of the protein matrix, particularly when sulfur dioxide was present, was directly related to the ease of milling and starch separation. Other workers (5,6) have employed the sectioning procedure to determine the degree of steeping by visually examining the extent of starch loss from sections. Steeping whole kernels for this purpose has the disadvantage that one cannot know the exact composition of steeping medium in direct contact with endosperm inside the kernel. It is known that

[1]Manuscript received May 5, 1960. Presented at the 45th annual meeting, Chicago, Ill., May 1960.

both the external steeping medium and the water phase inside the kernels continually change in pH and sulfur dioxide content for a number of hours after steeping is started[2].

To steep endosperm under accurately known conditions, thin sections of endosperm were cut from water-softened corn kernels and then steeped in a large excess of medium. Extent of steeping was determined by measuring the increased transmission of light through the section resulting from dislodgement of starch granules. Horny endosperm was used because its starch granules are embedded in relatively thick protein matrix and it therefore presents the greatest difficulty in starch extraction. Section thickness was set at 10μ, because starch granules in the horny endosperm average about 10μ in diameter but the endosperm cells have dimensions of 50 by 100μ (10). These sections, therefore, could offer little resistance to passage of water or chemicals into their interiors. The starch granules were retained in the sections only by the strength of the protein matrix, because in a 10μ section no endosperm cell could remain unopened.

With this technique we have studied the effects of sulfur dioxide concentration and steeping temperature upon the rate and extent of starch release from horny endosperms of several varieties of corn and of corn that was artificially dried.

Materials and Methods

Corn Samples. The sources of corn samples used in this work are given in Table I. The first two yellow-dent hybrid corns are typical of those found in commercial trade channels and are used as reference grains in this laboratory. One of them, CPC-50, is of average

TABLE I

IDENTIFICATION OF CORN SAMPLES

CPC Number	Type	Location Grown	Drying Conditions	Protein
				% *d.b.*
50	Yellow dent hybrid	Justice, Ill.	104°F. (40°C.)	9.3
3	Yellow dent hybrid	Clinton, Ill.	104°F. (40°C.)	10.5
80	Yellow flint variety	Argentina	Field-dried	12.1
11	High-protein yellow dent hybrid[a]	Argo, Ill.	Field-dried	12.6
65	Yellow dent hybrid[b]	Urbana, Ill.	120°F. (49°C.)	11.7
62	Yellow dent hybrid[b]	Urbana, Ill.	180°F. (82°C.)	11.8
78	Yellow dent hybrid[b]	Urbana, Ill.	200°F. (93°C.)	11.7

[a] Experimental hybrid derived from Illinois high-protein strain by Funk Brothers Seed Co., Bloomington, Illinois.

[b] Picked at 32% moisture, dried to 11% moisture in air at 40% relative humidity and the indicated temperature. From the 1952 crop. (See reference 3.)

[2] Watson, S. A., and Hirata, Y., unpublished data.

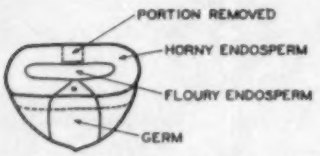

Fig. 1. Sketch of decrowned corn kernel showing approximate location of endosperm cube removed for sectioning.

hardness and protein content and the other one, CPC-3, is harder and of higher protein content than average dent corn. Argentine flint corn and the high-protein hybrid corn were included because of expected differences in endosperm properties. Three samples of yellow dent corn artificially dried by the Agricultural Experiment Station, University of Illinois, were included to study the steeping properties of heat-damaged corn.

Preparation of Sections. Eight intact kernels of each sample were allowed to sorb moisture from a saturated atmosphere for 24 to 48 hours. A cube of horny endosperm measuring about 3 mm. on each side was cut from the abgerminal side of each kernel as shown in Fig. 1. Sections of 10μ thickness were cut from each cube on a freezing stage mounted on a Leitz rotary microtome. About five sections were cut and discarded to avoid the aleurone and peripheral endosperm layers; the next five sections, after cutting, were held in water to await steeping. Holding time in water was never more than 2 hours and this did not influence results.

Steeping. The steeping vessels were 2-oz. screw-cap bottles. Twenty-five milliliters of the steeping medium were put in the bottle with five intact sections from a single kernel. Air was displaced with nitrogen to minimize oxidation of sulfur dioxide and the consequent drop in pH of the medium. Steeping media were solutions of potassium metabisulfite in distilled water at concentrations equivalent to 0.05 to 0.4% sulfur dioxide as determined by iodine titration. Initial pH of the potassium metabisulfite solution was 4.0 and it did not drop below 3.6 in any experiment. Bottles containing the sections, fastened in a reciprocating shaker immersed in a constant-temperature water bath, were shaken continuously at a rate of 80 (2-in.) strokes a minute. Temperature of most experiments was 52°C., but several runs were made at 60°C. Eight bottles each containing five sections from a single kernel were used in each experiment. Occasionally sections were discarded if they curled, fragmented, or produced results completely out of line, but data presented include readings from no less than six kernels.

Photometric Estimation of Starch Release. To score sections for starch release, one section from each of the eight kernels was removed at time intervals of 0.5, 1, 2, 4, and 6 hours. Each section was then placed on a microscope slide and covered with a drop of dilute iodine-potassium iodide solution (about 0.005*N* I_2). After several minutes, when the starch granules had stained dark blue, the iodine solution was replaced with glycerine and the section covered with a glass slip. The section was centered and focused under a 32-mm. (6×) objective lens of the microscope equipped with a 20× ocular lens. Amount of light transmitted was measured with a photocell-galvanometer combination. Light intensity was adjusted by positioning the substage condenser to give a reading of 10 ma. through the glass slide with the section moved out of the field. Readings were recorded as percent light transmitted by the sections.

Several attempts were made to calibrate the transmission readings in terms of actual starch released. Individual sections and steepwater from sections were analyzed for starch by a microcolorimetric iodine method. Results by this method correlated positively with the transmission method results, but the sensitivity of the iodine method was too low to validate completely the other method. A more positive check on the results by the transmission method was provided by running a blank section from which all starch had been removed. Cox, MacMasters, and Hilbert (1) found that, when a section is covered with 70% sulfuric acid for 1 minute, all starch granules are released but protein matrix is not altered. This procedure was applied to sections of the corn samples employed in this work. These values are considered to be maximum transmission values obtainable by steeping and are therefore recorded on the graphs for comparison with readings of steeped sections. Maximum transmission values of all steeped sections were below the values for the sulfuric acid-treated sections. This was due to occasional islands of starch granules that still remained after the 6-hour shaking period. Since most curves were still ascending at the sixth hour, these pieces of endosperm should eventually have been shaken loose.

Sections shaken in distilled water for 6 to 24 hours at 52°C. gave zero light transmission. Several sections treated with alkaline detergent to remove all starch and protein, leaving only the cell-wall network, gave 97.5% transmission. The transmission value of sulfuric acid-destarched sections was about 90% for ordinary dent corn but was only 65% for the high-protein corn.

Results and Discussion

When light-transmission values of steeped sections of individual kernels were plotted against time of steeping, families of curves were obtained as shown in Figs. 2 and 3. The dent corn used in the drying studies, CPC-65, showed rather good agreement among individual kernels, but the reference dent corn, CPC-3, exhibited considerable variation among kernels (Fig. 3). Other samples fell between these extremes. Curves shown in Figs. 4 to 8 are constructed from the average transmission values for sections of the eight kernels at each time interval. Use of a larger number of kernels in each experiment was impractical, so statistical evaluation was used as an aid in data interpretation. The data were subjected to analysis of variance, and least significant difference (LSD) values at the 5% level were calculated. It is assumed that variances of each group of eight samples are similar, since the kernels were taken from the same populations. Differences between any two points smaller than the LSD are not significantly different at the 5% level.

The results obtained with horny endosperm sections from the two samples of ordinary yellow dent corn are given in Figs. 4 and 5. The sample, used for Fig. 4 (CPC-50) is representative of average commercial corn. The greatest rate and extent of starch release were ob-

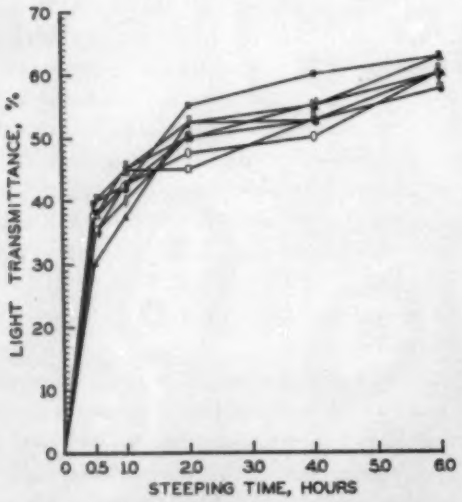

Fig. 2. Reproducibility of starch release from 10-μ sections of horny endosperm of eight kernels of yellow dent corn, CPC-65.

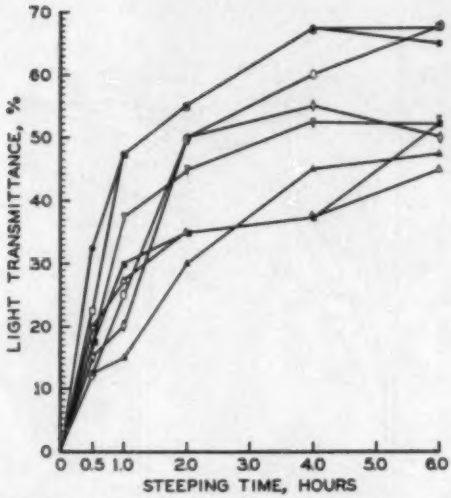

Fig. 3. Reproducibility of starch release from 10-μ sections of horny endosperm of eight kernels of yellow dent corn, CPC-3.

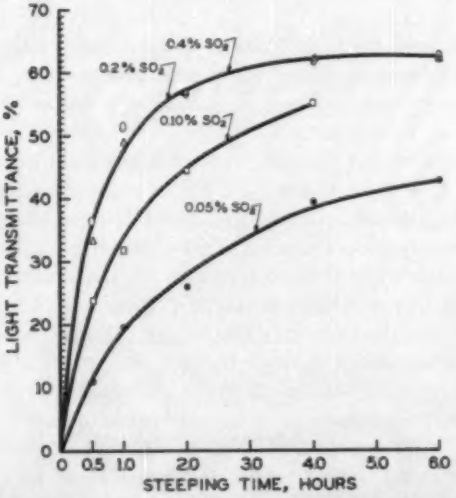

Fig. 4. Average starch-release rate from 10-μ sections of horny endosperm of yellow dent corn, CPC-50. LSD (5% level) = 6.8%. Starch-free transmittance = 91%. Steep temperature, 52°C.

tained with 0.2% sulfur dioxide. Although a run was not made using 0.15% sulfur dioxide, it is probable that the curve would have been

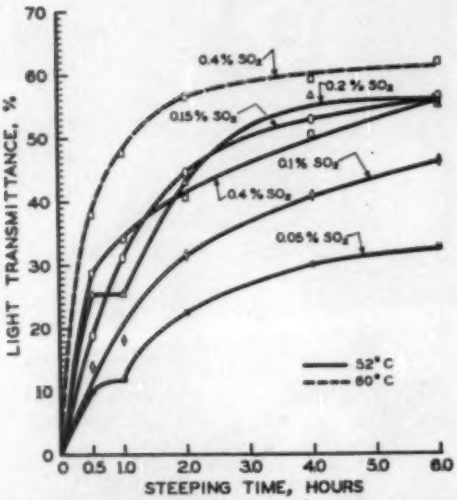

Fig. 5. Average starch-release rates from 10-μ sections of horny endosperm of yellow dent corn, CPC-3. LSD (5% level) = 1.3%. Starch-free transmittance = 87.5%. Steep temperatures: 52°C., solid lines; 60°C., broken lines.

similar to the curve for 0.20% sulfur dioxide since this was the case in Fig. 5. Comparison of these two curves indicates that CPC-3 horny endosperm is somewhat more resistant to the action of sulfur dioxide in releasing starch than CPC-50. This confirms previous data that CPC-3 has a harder kernel (7). Rate and extent of starch release are lower for CPC-3 than for CPC-50 at equivalent sulfur dioxide concentrations. Highest rate and greatest extent of starch release for CPC-3 were obtained on steeping at 60°C. with 0.4% sulfur dioxide, but a 0.2% sulfur dioxide concentration probably would have given a similar curve. No evidence of starch damage, as judged by loss of birefringence, was observed in those sections steeped at 60°C.

Flint corn is so named because the kernel appears to be very hard, owing to a larger proportion of horny endosperm than is found in dent corn. The floury portion of flint corn endosperm is entirely encased in horny endosperm. The data in Fig. 6 show that 0.2% sulfur dioxide effects starch release from Argentine flint horny endosperm sections comparable to that obtained with sections of the reference yellow dent corn, CPC-50 (Fig. 4). At sulfur dioxide concentrations below 0.2%, the Argentine flint endosperm gave lower rate and extent of starch release than the dent corns. This suggests that its horny endosperm contains thicker strands of protein matrix that require a

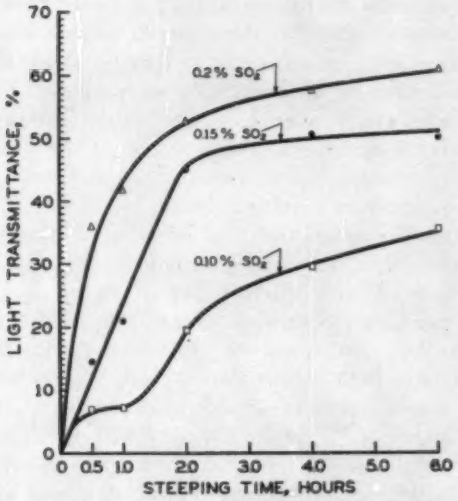

Fig. 6. Average starch-release rates from 10-μ sections of horny endosperm of Argentine flint corn, CPC-80. LSD (5% level) = 3.1%. Steep temperature 52°C. Starch-free transmittance, 86%.

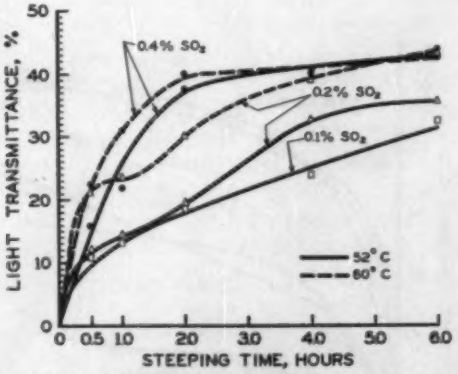

Fig. 7. Average starch-release rates from 10-μ sections of horny endosperm of high-protein hybrid dent corn, CPC-11. LSD (5% level) = 2.7%. Steep temperatures: 52°C., solid line; 60°C., broken line. Starch-free transmittance = 65%.

higher concentration of sulfur dioxide for maximum starch release.

The starch release from Argentine flint endosperm at 0.10% sulfur dioxide shows a pronounced lag between 0.5 and 1 hour (Fig. 6). This might be explained as release of loosely held granules during the first half-hour of steeping. These granules may have been some

that were cut with the microtome knife and hence were not entirely encased in protein matrix. For the granules entirely encased in protein matrix, there may be an induction period of about 1 hour for the low concentration of sulfur dioxide to react with the protein to an extent sufficient to weaken it so the starch granules will be released. There is no direct evidence that this is the correct explanation, but this same trend can be seen in the other two curves in Fig. 6 and on some curves in the other figures.

Figure 7 gives the results obtained with horny endosperm sections of high-protein yellow dent corn. A much restricted starch-release picture was obtained with this corn at all sulfur dioxide levels as compared with ordinary dent corn. One parent of this hybrid is the Illinois high-protein strain of corn (9), which carries the trait of a horny endosperm of high density due to thick protein matrix strands. The greatest rate and extent of starch release were obtained with 0.4% sulfur dioxide at 52° and 60°C. and with 0.2% sulfur dioxide at 60°C. However, with the latter, the rate up to 2 hours was lower than at 0.4% sulfur dioxide because of a lag at 1 hour similar to that obtained with the flint corn. The higher temperature apparently enhanced the action of sulfur dioxide at the 0.2% level.

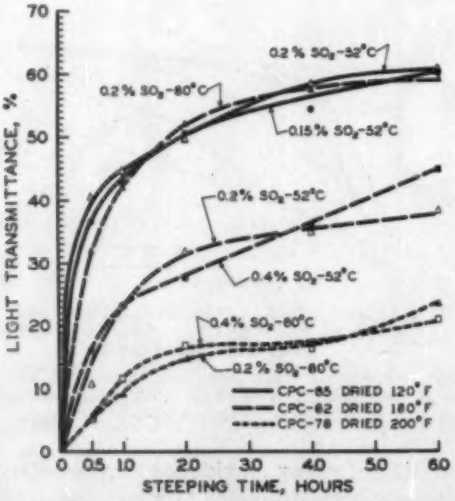

Fig. 8. Average starch-release rates from 10-μ sections of horny endosperm of yellow dent corn dried at three temperatures; CPC-65 dried at 120°F. (solid line); CPC-62 dried at 180°F. (broken line); CPC-78 dried at 200°F. (dashed line). Steep temperatures 52° and 60°C. LSD (5% level) = 2.5%. Starch-free transmittance = 88%.

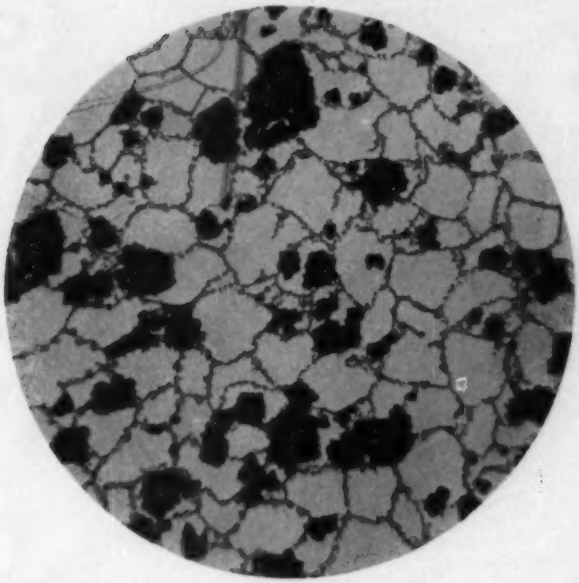

Fig. 9. Section of horny endosperm of dent corn, CPC-62, dried at 120°F. (49°C.) after 4 hours of steeping at 0.15% SO_2, 52°C. Light transmission, 65%. Mag. 44×.

Effect of Conditions of Artificial Drying. The use of excessive temperatures in the artificial drying of corn causes decreased yield and quality of starch in the wet-milling process (2,3). The swelling of protein matrix observed by Cox, MacMasters, and Hilbert (1) with endosperm sections of normal corn steeped in sulfur dioxide was not observed by them with corn dried at high temperatures.

The data in Fig. 8 confirm this effect, using our technique with artificially dried corn. The corn was dried from 32 to 12% moisture with air at 40% relative humidity and at temperatures of 120°, 180°, and 200°F. (49°, 82°, and 93°C.). These samples were dried in a farm-scale batch dryer (2) and are part of the same lots used by MacMasters *et al.* (3) from the 1952 crop year. The corn dried at 120°F. (49°C.) gave data (Fig. 8) similar to the reference dent corn dried at 104°F. (40°C.) (Fig. 5). The endosperm sections of corn dried at 180°F. (82°C.) released about two-thirds as much starch as the control when steeped at 52°C. Steeping at 60°C. with 0.2% sulfur dioxide, however, apparently overcame the damaging effect of the drying at this temperature. Corn dried at 200°F. (93°C.) gave only one-third the extent of starch release obtained with the control

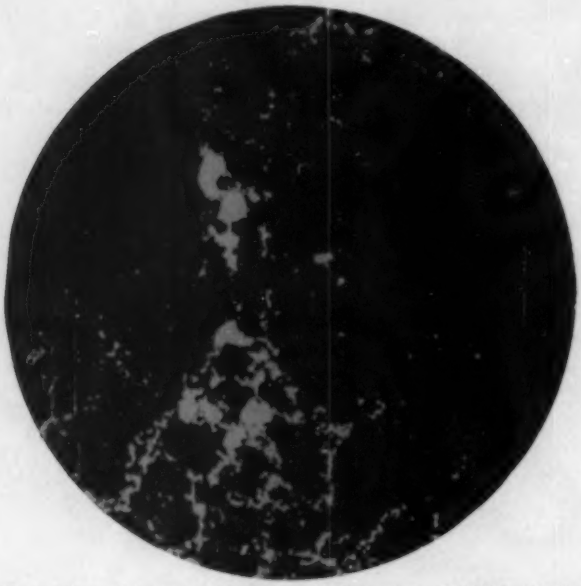

Fig. 10. Section of horny endosperm of dent corn, CPC-78, dried at 200°F. (93°C.) after 6 hours of steeping at 0.4% SO_2, 60°C. Light transmission 15%. Mag. 35×.

even with steeping at 60°C. with either 0.2 or 0.4% sulfur dioxide. The transmission of 15% recorded for these sections was due to loss of pieces of intact endosperm, and it is doubtful that individual starch granules were released from protein matrix. Cracking and crazing of endosperm in the samples dried at the two higher temperatures made the preparation of intact sections very difficult.

The inability to release starch from corn dried at high temperature is dramatically illustrated in Figs. 9 and 10. The first photograph shows the normal starch release obtained with the corn dried at 120°F. (49°C.). The second shows the almost complete retention of starch in the section of the corn dried at 200°F. (93°C.) even though it was steeped under drastic conditions.

It is evident that the use of a temperature of 200°F. (93°C.) for artificially drying corn damages endosperm by irreversibly locking the starch granules into the protein matrix. This observation explains the inability of the wet-miller to extract starch from horny endosperm. Starch obtained from such badly damaged corn comes only from the floury endosperm portion of the kernel and carries a high

protein content due to pieces of protein that adhere to starch granules. The horny endosperm cannot be ground into pieces small enough to pass through the screens used for fiber removal and therefore must pass into the by-product cattle feed. Starch in feed has about one-sixth the monetary value that it has as purified starch.

The use of a 60°C. steeping temperature gave normal starch release from the corn dried at 180°F. (82°C.). However, the countercurrent manner used with batteries of commercial corn steeps makes impossible the use of a temperature above 55°C. or a sulfur dioxide concentration higher than about 0.2%; the delicate balance of lactic acid fermentation necessary for optimum steeping would be destroyed.

It is obvious that the full commercial value of corn cannot be realized by processors unless farmers and receiving terminal elevator operators adhere strictly to moderate drying conditions.

Acknowledgment

The co-operation of J. H. Ramser, Department of Agricultural Engineering, and G. H. Dungan, Department of Agronomy (retired), of the University of Illinois for supplying samples of artificially dried corn is gratefully acknowledged.

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STRUCTURE OF THE STARCH GRANULE

II. Action of Various Amylases on Granular Starches[1]

HARRY W. LEACH[2] AND THOMAS J. SCHOCH[2]

ABSTRACT

The dissolving action of various amylases on granular starches has been studied, with particular attention to bacterial alpha-amylase. The following starches are listed in order of increasing resistance to amylase: waxy maize, tapioca, waxy sorghum, sorghum, corn, wheat, rice, sago, arrowroot, potato, heat-moisture-treated potato, high-linear corn. Two general patterns of enzymatic solubilization were observed: A, extensive erosion and fragmentation of corn and sorghum starches and their waxy counterparts, and B, selective granule-by-granule destruction of potato and most of the other starches. Even after 50% solubilization of corn and sorghum starches, the fragmented residues have iodine affinities, intrinsic viscosities, Brabender viscosities, and swelling and solubility patterns similar to those of the parent starches. It is suggested that these cereal starches may have a porous granule structure accessible to the enzyme, while potato starch is less permeable. Other evidence indicates that the starch granule has no restrictive outer membrane, that the starch fractions are distributed uniformly throughout the granule, and that amylase action is not related to considerations of molecular association or crystallization pattern.

A previous publication from this laboratory described the patterns of progressive swelling and solubilization of various granular starches when heated in water (7). The results were indicative of the extent and strength of molecular association (i.e., hydrogen bonding) within the granule. Recent work by Sandstedt (9) and by Badenhuizen (2) suggests that the dissolving action of amylases on granular starches may offer another means for studying granule structure. The extent and mode of enzyme digestion of various starches might provide evidence of physical differences. In addition, comparison of the undissolved residue with the original granular starch might indicate the nature of any skeletal structure.

Materials and Methods

Sources of Starches.

Corn, sorghum, and waxy sorghum ("white milo"), Corn Products Co.

Waxy maize ("Amioca" brand), American Maize-Products Co.

Wheat, Huron Milling Division, Hercules Powder Co.

[1] Manuscript received March 17, 1960. Presented at the 45th annual meeting, Chicago, Illinois, May 4, 1960.

[2] George M. Moffett Research Laboratory, Corn Products Company, Argo, Ill.

Tapioca ("Maris" brand) and rice, Stein Hall & Co.

Potato, Northern Aroostook Starch Co.

Arrowroot, prime St. Vincent, Morningstar-Paisley, Inc.

Unbleached sago flour (kindly supplied by National Starch and Chemical Corp.) was suspended in water, screened through fine bolting cloth, and the starch filtered and air-dried.

High-linear corn starch, phenotype aeae, laboratory-prepared, 11.9% iodine affinity.

Sources of Enzymes.

Bacterial alpha-amylase, analytical grade, SKB value at 30°C. = 16,000, Wallerstein Co., Inc.

"Rapidase" brand bacterial alpha-amylase, SKB value at 30°C. = 10,000, Wallerstein.

Glucamylase, laboratory-prepared fungal amylase from *Aspergillus niger* (12).

Pancreatin, Takamine Laboratory, division of Miles Laboratories, Inc.

Malt diastase, analytical grade, activity = 1,000°L, Wallerstein.

Fungal alpha-amylase, analytical grade, SKB value at 30°C. = 10,000, Wallerstein.

"Mylase" SC brand fungal alpha-amylase from *Aspergillus oryzae*, SKB value = 6,000, Wallerstein.

Beta-amylase, analytical grade, activity = 1,000° L, Wallerstein.

Method of Digestion. The specified amount of enzyme powder (expressed as percentage of dry starch) was added to 400 ml. of an aqueous slurry containing exactly 100 g. of starch (on dry basis). Two milliliters of toluene were added to prevent bacterial or mold action. The slurry was then adjusted to the optimum pH level for the particular enzyme (see Table I), placed in a thermostatted water bath at the desired temperature, and slowly stirred for 24 hours. At the end of this period the mixture was cooled, adjusted to pH 3.0 with hydrochloric acid, and stirred for 15 minutes to inactivate the enzyme. The slurry was then adjusted to pH 6.0 and filtered on a Buchner funnel; the cake was thoroughly washed with distilled water. After air-drying at room temperature, the weight and moisture content of this residue starch were determined, and the percentage of solubilized starch calculated by difference.

Analytical Methods. Intrinsic viscosities (in 1*N* potassium hydroxide) and iodine affinities were determined on the original and enzyme-treated starches by the methods of Lansky *et al.* (6). Brabender viscosities were determined by cooking for 1 hour at 95°C., then cooling to 50°C. and holding 1 hour (8). Swelling and solu-

bility patterns were determined by the method of Leach *et al.* (7). Granule densities were determined in xylene at 30°C., using starches predried for 4 hours in the vacuum oven at 120°C. As an index of precision, average deviation of all density results was ± 0.0049.

Results

Comparative Activities of Various Enzymes. Preliminary tests were run to select the enzyme and digestion conditions which would give the desired solubilization of granular starch. Corn and potato starches were used as substrates, because they exhibit very wide differences in physical properties and granule organization. Table I shows the percentage of starch solubilized during 24 hours' digestion by each of the enzymes at the specified temperature and enzyme level. At 30°C., corn starch is solubilized to approximately the same extent (i.e., 18-22%) by 1% of bacterial alpha-amylase, pancreatin, glucamylase, or malt diastase. At 50°C. and 0.5% enzyme, the bacterial alpha-amylase is clearly the most active. The beta-amylase and fungal alpha-amylase showed low activity at 30°C. and were pre-

TABLE I

DIGESTION OF GRANULAR CORN AND POTATO STARCHES BY VARIOUS ENZYMES

ENZYME	pH	STARCH	ENZYME DOSAGE[a]	TEMPERATURE	STARCH SOLUBILIZED
			%	°C	%
Bacterial alpha-amylase	6.5	Corn	1.0	30	20.5
		Corn	0.5	50	49.5
		Potato	1.0	50	21.8
"Rapidase"	6.5	Corn	1.0	30	21.5
		Corn	0.5	50	48.6
Glucamylase	4.5	Corn	1.0	30	18.4
		Corn	0.5	50	36.4
		Potato	3.0	50	0.5
Pancreatin	7.0	Corn	1.0	30	20.7
		Corn	0.5	50	29.9
		Potato	1.0	50	12.5
Malt diastase	4.5	Corn	1.0	30	18.4
		Corn	0.5	50	6.0
		Potato	1.0	50	3.5
Fungal alpha-amylase	5.0	Corn	1.0	30	8.3
		Corn	0.5	50	4.9
		Potato	1.0	50	0.8
"Mylase" 5C	5.0	Corn	1.0	30	8.4
		Corn	0.5	50	6.7
		Potato	1.0	50	1.5
Beta-amylase	4.5	Corn	1.0	30	8.3
		Corn	0.5	50	0.0

[a] Enzyme dosage calculated on dry starch basis.

sumably inactivated at 50°C.

In all cases (and particularly with glucamylase), granular potato starch is very much more resistant to enzyme action than is corn starch. Similar conclusions have been reached by Balls and Schwimmer (3), by Gates and Sandstedt (5), and by Badenhuizen (2). The greater digestibility of corn starch by enzyme appears to be an inherent property of this starch, and is not due to granule damage caused by drying the corn or by factory milling. As evidence, the following two starches showed substantially the same susceptibility toward bacterial alpha-amylase as commercial corn starch: 1) laboratory-prepared starch from dough-stage dent corn (35% moisture) used directly without drying, and 2) prime corn starch from the floury endosperm, obtained from the factory stream prior to buhr-milling. In contrast, Alsberg and Griffing (1) and more recently Blish, Sandstedt, and Kneen (4) have attributed differences in the enzyme susceptibilities of wheat flours in large part to granule damage during dry-milling.

The soluble fractions from the various 50°C. conversions of corn starch were qualitatively chromatographed to indicate the probable identity of the sugar products. Bacterial and pancreatic amylases gave principally d-glucose, with decreasing amounts of maltose, maltotriose, and maltotetraose. Glucamylase gave only d-glucose, and malt diastase gave principally maltose with a trace of the triose. The fungal alpha-amylases gave only d-glucose, probably indicating the presence of glucamylase.

Conditions for Digestion with Bacterial Alpha-Amylase. Since the analytical grade of bacterial alpha-amylase had the highest solubilizing action on both corn and potato starches, further studies were made to establish the optimum temperature, digestion time, and enzyme level. The solubilization of both corn and potato starches increased as the temperature was raised from 30° to 50°C.:

Digestion temperature, °C.	*Starch Solubilized*	
	Corn starch +0.5% enzyme	*Potato starch +3% enzyme*
	%	%
30	17.2	4.7
40	28.1	12.6
50	49.5	29.7

At 50°C. neither starch showed any evidence of granule swelling, or any enlargement or loss of birefringence at the hilum. As shown in Fig. 1, both starches were solubilized at a rapid rate during the

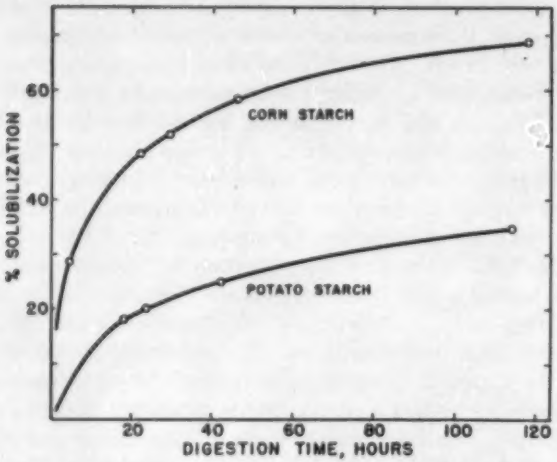

Fig. 1. Rate of solubilization of granular corn and potato starches by 0.5% bacterial alpha-amylase at 50°C.

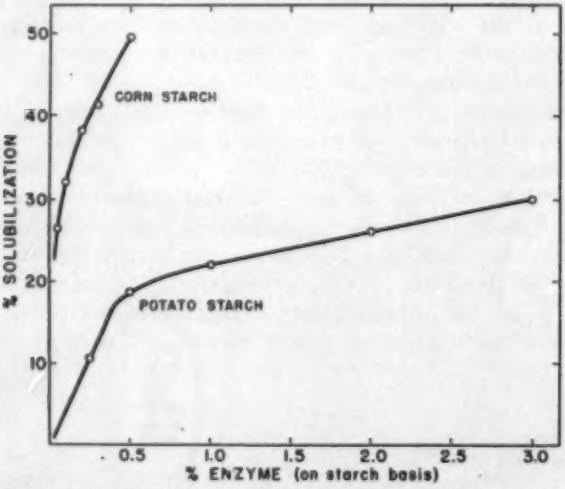

Fig. 2. Effect of dosage of bacterial alpha-amylase on the solubilization of granular corn and potato starches (24 hours' digestion at 50°C.).

initial 24 hours, and more slowly thereafter. The decreased rate of digestion after 24 hours may be due partly to enzyme inactivation and partly to inhibition by the sugar products. For example, 0.2% bacterial alpha-amylase at 50°C. solubilized 100 g. of corn starch

to the extent of 38.1% in 24 hours; a similar run in the presence of 30 g. of added d-glucose decreased the solubilization to 27.1%. In Fig. 2, 0.5% enzyme appeared to be an efficient level for solubilization of potato starch. Hence subsequent studies on various granular starches were conducted with 0.5% bacterial alpha-amylase (analytical grade) for 24 hours at 50°C.

Comparative Susceptibility of Various Starches. Eleven different unmodified starches were digested with bacterial alpha-amylase under the above specified conditions (i.e., 24 hours, 50°C., 0.5% enzyme). Also included was heat-moisture-treated potato starch, prepared by refluxing unmodified potato starch in boiling 70% diacetone alcohol for 4 hours (7). Table II lists the various starches in order of increasing resistance to alpha-amylase action. Waxy maize is the most susceptible (57.8% solubilized in 24 hours), and heat-moisture-treated potato starch and high-amylose corn starch are least attacked (15.5 and 14.0%, respectively). In no case was there any preferential attack on either the linear or the branched starch fraction, since the iodine affinity of each residue starch was substantially the same as that of the parent starch. In most instances, there was some decrease in both intrinsic viscosity and Brabender viscosity; however, this effect is considered relatively minor since all the residue starches are still essentially "thick-boiling" in character.

Neither corn nor potato starch appears to adsorb any bacterial alpha-amylase from aqueous solution, and hence their different susceptibilities cannot be ascribed to different adsorptive affinities for the enzyme. For example, 40 g. of corn starch were stirred for 30 minutes in 200 ml. of bacterial alpha-amylase solution at room temperature and pH 6.5. At enzyme concentrations in the range of 0.5 to 4.0 mg. per ml., the alpha-amylase activity of the aqueous substrate was not changed by contact with the starch, as determined spectrophotometrically by a modified Wohlgemuth assay. In a similar series of experiments with potato starch, the activity of the substrate was markedly increased by contact with the starch. This is due to the fact that granular potato starch preferentially adsorbs water from the surrounding enzyme solution, thus increasing the concentration or apparent activity of the latter.

Successive Enzyme Digestions. It was of interest to compare the residues from corn and potato starches at equally high levels of digestion. Corn starch was given three successive 24-hour digestions with 0.1% bacterial alpha-amylase at 50°C.; after each digestion the residue starch was filtered and dried to determine yield, then resuspended and treated with fresh enzyme. To compensate for its lower

TABLE II

SOLUBILIZATION OF VARIOUS GRANULAR STARCHES WITH BACTERIAL ALPHA-AMYLASE, AND PROPERTIES OF THE RESIDUE STARCHES
(0.5% enzyme, 24 hours' digestion, 50°C.)

STARCH	STARCH SOLUBILIZED	SAMPLE	INTRINSIC VISCOSITY	IODINE AFFINITY	DENSITY	BRABENDER VISCOSITY [a]			
						Concentration	At Peak	At 95°C.	At 50°C.
	%			%		g/500 ml	B.u.	B.u.	B.u.
Waxy maize	57.8	Untreated	1.58	....	1.480	30	760	160	220
		Residue	1.36	....	1.516	30	305	65	120
Tapioca	55.7	Untreated	2.03	3.15	1.521	30	590	150	290
		Residue	1.86	3.20	1.528	30	370	120	225
Waxy sorghum	55.7	Untreated	1.43	....	1.490	30	545	120	180
		Residue	1.33	....	1.522	30	205	45	85
Sorghum	50.2	Untreated	1.43	4.90	1.500	40	665	490	710
		Residue	1.31	4.97	1.504	40	375	230	440
Corn	49.5	Untreated	1.61	4.83	1.517	35	485	280	815
		Residue	1.51	4.72	1.511	35	370	180	545
Wheat	48.5	Untreated	1.25	5.30	1.542	55	530	145	>1000
		Residue	1.14	5.30	1.523	55	715	310	>1000
Rice	32.6	Untreated	1.42	4.70	1.510	40	670	735	>1000
		Residue	1.40	5.07	1.489	40	690	700	>1000
Sago	27.0	Untreated	1.60	4.98	1.494	35	590	190	420
		Residue	1.60	5.07	1.533	35	580	175	480
Arrowroot	20.8	Untreated	1.85	4.45	1.510	35	885	470	780
		Residue	1.85	4.65	1.526	35	850	530	960
Potato	18.4	Untreated	2.44	4.42	1.511	30	>1000	240	470
		Residue	1.53	4.65	1.541	30	>1000	180	310
Heat-moisture-treated potato	15.5	Untreated	1.86	4.45	1.507	35	>1000	750	930
		Residue	0.86	4.40	1.526	35	>1000	570	835
High-linear corn	14.0	Untreated	1.09	11.9					
		Residue	....	11.3					

[a] Brabender viscosities are given at the pasting peak, after holding 1 hour at 95°C., and after cooling and then holding 1 hour at 50°C.

TABLE III

PROPERTIES OF THE RESIDUES FROM CORN AND POTATO STARCHES AFTER EXTENSIVE SOLUBILIZATION WITH BACTERIAL ALPHA-AMYLASE

STARCH	SOLUBLES	INTRINSIC VISCOSITY	IODINE AFFINITY	BRABENDER VISCOSITIES[a]		
				AT PEAK	AT 95°C.	AT 50°C.
	%		%	*B* u	*B* u	*B* u
Corn						
Original	....	1.61	4.83	500	330	830
Enzyme-digested	58.0	1.47	4.55	375	250	530
Potato						
Original	....	2.44	4.42	>1000	240	470
Enzyme-digested	56.6	1.70	4.85	>1000	260	405

[a] Brabender viscosities were run at 35 g. per 500 ml. on the corn starch samples, and at 30 g. on the potato starch samples.

susceptibility, potato starch was given four successive digestions, and the enzyme level was increased to 1.0%. Results are shown in Table III. While Brabender viscosities and intrinsic viscosities are somewhat lower, the residues from both starches are still considered as essentially thick-boiling, even after conversion of 56–58% of the original starch substance.

Microscopic Examination. As observed under the polarizing microscope, the various starches show two distinctly different patterns of enzyme attack: A, extensive granule erosion and fragmentation with corn and sorghum starches and their waxy counterparts, and B, selective-granule digestion with potato and the other starches studied. (Wheat starch is intermediate in behavior.) In the typical case of corn starch, the enzyme appears to attack the hilum first, with the loss of birefringence in that region. Schwimmer (11) has likewise observed this type of enzyme action on granular corn and wheat starches. The hilum appears to be the most susceptible part of the granule, since it is attacked in the early stages of enzyme digestion, and since it is likewise the first region to show loss of birefringence when the granule swells in hot water. As enzyme digestion proceeds most of the corn starch granules become highly eroded and fragmented (Fig. 3, A). However, there are always a few granules (i.e., 5–10% of the original starch) which show no evidence of any erosion, even after 50% solubilization. These resistant granules are of both the angular and the round types (i.e., from the horny and floury endosperm, respectively).

In contrast, potato starch shows no evidence of granule erosion or decrease of birefringence even after 56% solubilization (Fig. 3, B). During enzyme digestion, the microscope will show an occasional

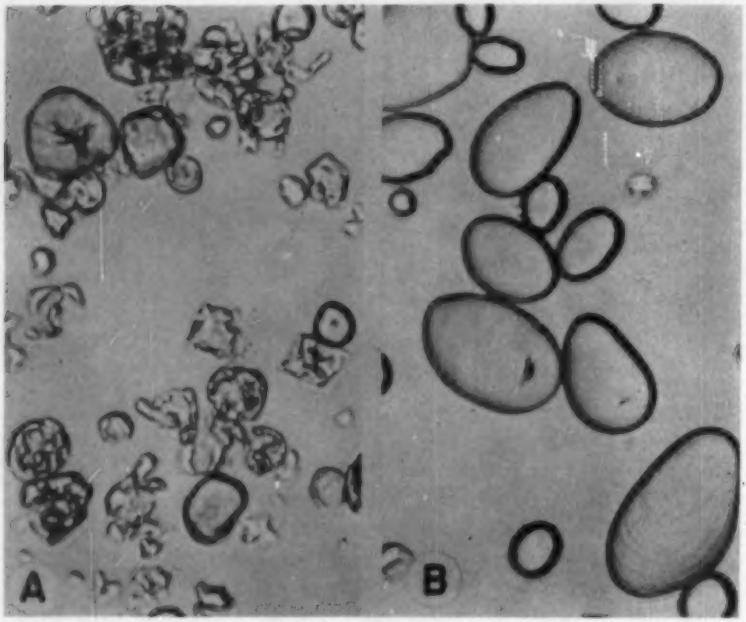

Fig. 3. Microscopic appearance of granular corn (A) and potato (B) starches after approximately 50% solubilization by bacterial alpha-amylase.

"ghost granule" undergoing rapid dissolution. Hence the enzyme attacks and completely destroys a few granules at a time, though the reason for this selectivity is not apparent. The enzyme shows no preference for either the large or the small starch granules. To prove this point, the parent potato starch and an enzyme-treated residue (namely, 56.6% solubilized by four successive digestions with 1% bacterial alpha-amylase) were each photomicrographed, and the granule diameters (i.e., average of long and short dimensions) were manually measured on approximately 400 granules of each sample. Size distribution curves were then obtained by plotting granule diameters against percentage frequency. The curves for the parent starch and the enzyme-digested residue were virtually identical, and the "number-average" and "weight-average" granule diameters (10) did not differ substantially:

	Number-Average Granule Diameter	*Weight-Average Granule Diameter*
	μ	μ
Untreated potato starch	20.9	37.1
Enzyme-digested residue	18.4	34.3

TABLE IV

SWELLING AND SOLUBILITY PATTERNS OF TREATED POTATO STARCHES

PASTING TEMPERATURE	SWELLING POWER[a]			SOLUBILITY		
	A Unmodified	B Enzyme Residue	C Water-Digested	A Unmodified	B Enzyme Residue	C Water-Digested
°C.	*times*	*times*	*times*	%	%	%
60	42.9	2.4	2.0	12.2	0.5	0.3
65	91.2	9.8	23.6	19.8	3.2	10.2
70	141.	26.3	36.1	28.0	6.8	11.9
75	200.	31.1	44.5	29.0	9.0	11.9
80	261.	41.8	61.5	34.3	10.0	15.1

[a] Corrected for solubility.

Effect of Exhaustive Drying on Enzyme Susceptibility. The enzyme susceptibility of potato starch is substantially increased by exhaustive drying. For example, the original potato starch gave 29.7% solubles when digested for 24 hours at 50°C. with 3% bacterial alpha-amylase. When the potato starch was dried for 16 hours *in vacuo* at 120°C., a similar enzyme treatment gave 49.7% solubles. This might perhaps be attributed to dextrinization or heat-damage during drying. However, when untreated potato starch was slowly dried to a moisture level of 0.27% by 10 weeks *in vacuo* over phosphorus pentoxide at room temperature, a similar enzyme treatment gave 54.5% solubles. Exhaustively dried starches develop considerable heat when contacted with water, which might conceivably cause damage or cracks within the granule. To avoid this, both of the above dried starches were exposed to laboratory air for several days to regain their equilibrium moisture contents, before treating with enzyme. No hydrolytic modification would be expected during drying, since the slurry pH of the original potato starch and the two dried samples was in the range of 6.7 to 6.8.

Heat-Moisture Treatment. Some concern was felt lest the enzyme resistance of potato starch might be due to the substantial heat-moisture modification incurred during prolonged digestion at 50°C. While four 24-hour digestions of potato starch at 50° C. cause only a minor change in the Brabender viscosity (Table III), nevertheless this amount of heat-moisture treatment does decrease the swelling and solubility of the starch. For example, swelling and solubility patterns were determined on the following three starches: A, unmodified potato starch; B, the residue after 56.6% solubilization of potato starch by four successive treatments with 1% bacterial alpha-amylase at 50°C.; C, potato starch after four similar digestions in water at 50°C. without enzyme. The two latter samples show con-

siderable reduction in both swelling power and solubility (Table IV). However, from a practical standpoint, it was necessary to use a digestion temperature of 50°C. in order to solubilize potato starch to a significant extent, since even 20% of bacterial alpha-amylase at 30°C. solubilized only 9.2% of the starch in 24 hours. While heat-moisture modification undoubtedly does occur at 50°C., this cannot possibly account for the enzyme resistance of potato starch, for two reasons. First, as previously mentioned, corn and potato starches still show major differences in solubilization when treated for 24 hours with bacterial alpha-amylase at 30°C. and at 40°C. Second, even though tapioca starch is very prone to undergo heat-moisture modification, it is still highly susceptible to enzyme attack.

Discussion and Conclusions

Certain general conclusions seem to be valid:

1. Enzyme susceptibility is not related to external surface area of the starch granules, in agreement with conclusions by Schwimmer (11). Thus the small-granule rice starch is in an intermediate position in Table II, and the other starches show no correlation between granule size and extent of solubilization.

2. The X-ray diffraction pattern of the starch has no relation to enzyme susceptibility. The starches tested are reputed to have the following spectra: wheat, rice, corn, sorghum, waxy maize, waxy sorghum, and heat-moisture-treated potato give an A-pattern; potato and high-linear corn starches give a B-pattern; arrowroot, tapioca, and sago give a C-pattern (2).

3. The concept of a peripheral membrane or hull enclosing the granule is untenable for corn and sorghum starches and their waxy counterparts. Even after extensive erosion and fragmentation, the residues still give Brabender viscosity curves typical of thick-boiling starches. As additional evidence for the absence of any membrane, swelling and solubility data were determined on the residue after 50% solubilization of corn starch by bacterial alpha-amylase. Despite the fissured and fragmented nature of the granules, this product showed even lower swelling and solubilization than the parent starch (Table V). However, the possibility of an enclosing membrane is not excluded with starches such as potato which are digested by selective-granule attack.

4. At least in the cases of corn and sorghum starches, the linear and branched fractions must be distributed uniformly throughout the granule. Even after half the total starch substance was solubilized,

TABLE V

EFFECT OF ENZYMATIC DIGESTION ON THE SWELLING POWER AND SOLUBILITY OF CORN STARCH

(Comparison of untreated corn starch and residue after 50% solubilization by 0.5% bacterial alpha-amylase for 24 hours at 50°C.)

PASTING TEMPERATURE	SWELLING POWER[a]		SOLUBILITY	
	Untreated	Residue	Untreated	Residue
°C	*times*	*times*	%	%
65	4.4	4.0	2.6	1.1
70	7.8	7.1	6.8	3.7
75	9.5	8.7	8.7	5.4
80	10.5	9.0	9.9	6.5
85	11.8	9.9	10.2	8.5
90	16.4	12.8	16.7	12.6
95	23.6	25.8	24.6	23.5

[a]Corrected for solubles.

the eroded and fragmented residues have substantially the same iodine affinities as the parent starches. This same evidence shows that the enzyme has no preference for either fraction. Since tapioca, arrowroot, sago, and potato starches are solubilized by attack on individual granules, no similar conclusion can be made regarding distribution of their fractions.

5. The waxy starches are somewhat more susceptible to enzyme attack than the corresponding normal starches. Also, high-linear corn starch is least affected by enzyme. Similarly, Schwimmer (11) found that normal pea starch is attacked more rapidly than the high-linear starch from wrinkled peas. Hence the presence of linear fraction probably retards solubilization by enzyme.

6. In general, intrinsic viscosities of the residues after enzyme attack are only slightly lower than the values for the parent starches. This is in contrast with the action of mineral acids on granular starches, which causes extensive hydrolytic breakdown throughout the granule. Hence it is presumed that the enzyme does not penetrate freely into the molecular lattice of the granule, but is limited to certain accessible surfaces or regions.

7. The absolute densities of the residues are very little different than the densities of the parent starches (Table II). This suggests that the enzyme does not preferentially digest the more amorphous or disorganized regions of the granule, since such action should cause a significant increase in density.

8. Enzyme susceptibility has no relationship to the patterns of swelling and solubilization of the starches when heated in water. Thus, weakly-bonded potato and tapioca starches swell freely to give stringy cohesive pastes, yet they show opposite susceptibilities toward

enzyme. Strongly bonded corn and heat-moisture-treated potato starches both show restricted swelling and give "short" heavy-bodied pastes, yet they differ widely in enzyme susceptibility.

Hence it is concluded that the enzyme susceptibility of granular starches is not influenced by physicochemical considerations of micellar structure, internal molecular association, or type of crystallinity. As Badenhuizen (2) has plausibly suggested, the more susceptible granules may possess pores or a coarse spongelike structure, with openings of sufficient size to admit the enzyme molecules. These pores must be characteristic of the particular species of starch, and not merely fissures produced during drying or manufacture. However, extreme drying (even without heat) increases the enzyme susceptibility of potato starch, perhaps by formation of a spongy internal structure.

Acknowledgment

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A COLORIMETRIC METHOD FOR DETERMINING FAT ACIDITY IN GRAIN[1]

DORIS BAKER[2]

ABSTRACT

A rapid colorimetric method for determining fat acidity in grain has been developed. The method is based upon the reaction of the fatty acids in benzene solution with aqueous cupric acetate to form soaps. The copper soaps are soluble in the benzene solution and the intensity of the resulting blue color of the solution is measured by a colorimeter. The intensity of the blue color measured as percent transmittance is compared with fat acidity values expressed as the number of milligrams of potassium hydroxide required to neutralize the free fatty acids in 100 grams of dry grain. For samples of wheat and corn the relationship between fat acidity values and percent transmittance is linear in the range of fat acidity values 20 to 100. The coefficient of correlation is −0.988 and −0.983, respectively, with standard deviations from regression of 2.47 and 3.42. The time required for the entire test is 10 minutes.

The fat acidity of grain is determined by extraction of the oil from the grain followed by titration of the free fatty acids present in the oil. In the method generally used for this determination (1), the oil is extracted from the grain with petroleum ether in a Soxhlet extractor and the titration is carried out in a solution of benzene-alcohol with standard aqueous potassium hydroxide solution. A rapid method (2) of similar principle has been proposed in which the extraction of the oil is simplified by using a grinder-extractor (Stein mill).[3] In both methods the fat acidity value is expressed as the number of milligrams of potassium hydroxide required to neutralize the free fatty acids in 100 g. of dry grain.

The new colorimetric method is based upon the reaction of fatty acids to form metallic soaps. Kaufman and Budwig (3) used cupric acetate to detect the spots of fatty acids on paper chromatograms. This paper reports an investigation of the reaction of cupric acetate with fatty acids dissolved in benzene. An aqueous solution of cupric acetate when shaken with fatty acids dissolved in benzene reacts with the fatty acids to form benzene-soluble copper salts which impart a blue color to the benzene solution. The reagent is sensitive enough to detect differences in the amounts of free fatty acids pres-

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[3] The mention of specific instruments is made for the purpose of identification and does not imply any endorsement by the United States Government.

ent in the oil from damaged grain. The differences in the smaller amounts of free fatty acids present in sound grain, whose fat acidity values are generally below 20, are not so easily detected. The intensity of the blue color in the benzene solution can be measured with an appropriate instrument. A comparative study was made of this colorimetric method with the usual procedure involving titration with standard potassium hydroxide solution.

Materials and Methods

Fat acidity was determined by the colorimetric method on 85 samples of wheat and 79 samples of corn. The transmittance readings were compared to the fat acidity values as determined by the method of the Association of Official Agricultural Chemists (1).

The percent transmittance of the solutions was measured with a Bausch & Lomb "Spectronic 20" spectrophotometer. As the fat acidity value increased, the intensity of the blue color increased and the percent transmittance decreased. Fatty acid solutions containing 10, 20, 30, 50, 80, and 100 mg. of fatty acids in 5 ml. benzene were treated with cupric acetate and transmittance curves from 350 to 700 $m\mu$ were made. From these curves the wave length of 640 $m\mu$ was chosen for measuring the percent transmittance of the copper soap solutions. At this wave length the transmittance of wheat and corn oil solutions in benzene is 98 to 100%.

A sample of grain weighing 40 g. is ground for 1 minute in a grinder-extractor (Stein mill). Then 50 ml. benzene are added to the ground sample and the oil is extracted by running the mill 4 minutes. The sample is filtered and 10 ml. of the filtrate measured into a test tube containing 2 ml. of 5% cupric acetate solution. The tube is stoppered and shaken by inverting rapidly 50 times by hand. After the solution has separated into two layers, the top benzene layer is decanted through a fluted filter into a colorimeter tube and the percent transmittance read at 640 $m\mu$ with a colorimeter or spectrophotometer. Benzene is used as the blank for adjusting the instrument.

Results and Discussion

Graphs of percent transmittance (Y) vs. fat acidity values (X) show a linear relationship in the range of fat acidity values 20 to 100. (See Figs. 1 and 2.) Above and below this range the relationship becomes curvilinear; therefore statistical analyses were confined to the samples within the range 20 to 100. Coefficients of regression and correlation for 60 samples of corn were −0.830 and −0.983, respective-

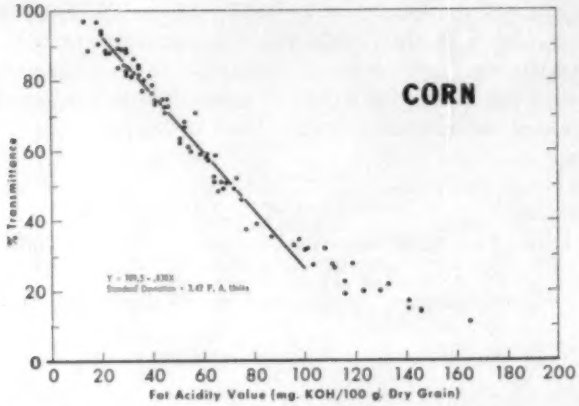

Fig. 1. Relation between fat acidity determined by the AOAC method and the percent transmittance at 640 mμ of benzene extracts of 79 corn samples after reaction with aqueous cupric acetate. (Y = 109.5−.830X; Standard Deviation = 3.42 F.A. units.)

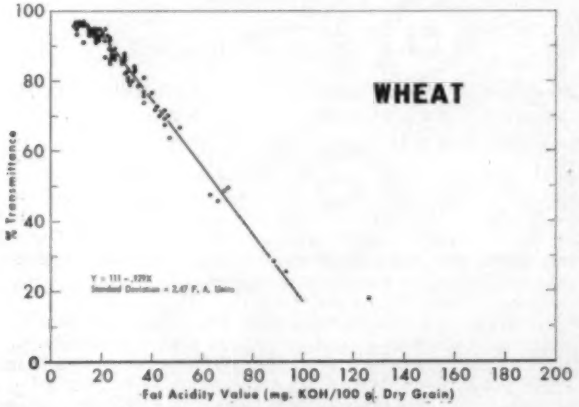

Fig. 2. Relation between fat acidity determined by the AOAC method and the percent transmittance at 640 mμ of benzene extracts of 85 wheat samples after reaction with aqueous cupric acetate. (Y = 111−.929X; Standard Deviation = 2.47 F.A. units.)

ly, with sample standard deviation from regression of 3.42 fat acidity units. For 54 samples of wheat, coefficients of regression and correlation were –0.929 and –0.988, respectively, with sample standard deviation from regression of 2.47 fat acidity units.

Since fat acidity values between 20 and 100 are significant in defining grain condition, the colorimetric method would be useful

where a rapid determination of fat acidity is needed. The entire test can be completed within 10 minutes. The regression lines of Figs. 1 and 2 may be used for converting transmittance readings to fat acidity values or for setting up a scale of transmittance readings indicating degrees of deterioration.

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OTTAWA MICRO FLOUR MILL[1]

J. G. Kemp, A. G. O. Whiteside, D. C. MacDonald, and H. Miller

ABSTRACT

A micro flour mill was built for the rapid milling of small samples of wheat. It consists of four stands of rolls with two breaks and two reductions. The roll units are superimposed upon each other with a bran bolter below the second break unit and a flour bolter located at the bottom of the mill. Each roll unit has a fixed roll and a spring-loaded adjustable roll. Four wheat samples of 100 g. or less may be milled at one time.

In preliminary trials free-milling hard wheats yielded about 60 g. of flour per 100 g. of wheat in 7 minutes' mill-run time. Ten minutes' mill-run time increased the yield to 65 g. or more. Softer wheats required 13 minutes for the higher flour yields. Milling losses were in the order of 3 to 4 g. Flour ash was about 0.48%. The effective mill capacity was 18.4 samples per hour for the 10-minute and 14.6 samples for the 13-minute mill-run schedules.

The need for producing flour from small samples of wheat for quality tests has been recognized for many years. Various methods ranging from the adaptation of burr and roll grinders with subsequent sieving operations (1,3,4,5) to the use of laboratory flour mills employing some of the basic operations of commercial mills (2,6) have been described in the literature.

[1] Manuscript received June 1, 1960. Contribution No. 1 from Engineering Research Service and No. 35 from the Genetics and Plant Breeding Research Institute, Research Branch, Canada Department of Agriculture, Ottawa.

A mill similar to the design of Shoup *et al.* (7) was constructed by R. H. Cunningham at the Research Station, Winnipeg. This was found useful in the development of the mill at Ottawa for the milling of small samples of wheat into flour for screening plant breeders' selections for quality in early generations and for use in genetic studies of quality components. The main considerations for the new mill were that it be designed to handle 100-g. samples or less with a minimum of manual labor, and that this operation should be accomplished as rapidly as possible in order that many samples can be milled in a short time. The extraction percentage and the grade of flour should be a good representative of the wheat sample.

Description and Construction

The mill consists of four stands or units of rolls with two break and two reduction pairs. These four units are superimposed upon each other, with a bran bolter below the second break unit and a flour bolter located at the bottom of the mill. The rolls are grooved to receive divider plates which divide the mill into sections to accommodate the milling of four samples of wheat at one time. This principle was used in the design of Shoup *et al.* (7). The wheat is fed to the first break unit at the top of the mill by the mechanical vibrator feeder. The mill, which weighs about 1,000 pounds, stands 66 in. high and measures 22 by 30 in. at the base and is powered by a 1½-h.p. motor. A view of the completed mill is shown in Fig. 1; Fig. 2 is a diagrammatic representation of the operational principles of the mill.[2]

The samples are fed from four individual stationary hoppers to a four-sectioned vibrator feeder (Fig. 3). The vibrator feeder is oscillated 1/32 in. at a rate of 2,000 cycles per minute. The feed rate can be adjusted to deliver a 100-g. sample from in 2 to 8 minutes.

The basic design and construction of each of the four roll units is identical. Figure 4 shows a partially assembled roll section. The 6-in.-diameter rolls are grooved for divider plates and each run has approximately a 1½-in. roll face. Each roll unit has a fixed roll and a spring-loaded adjustable roll.

To provide for parallel setting and clearance of the rolls, the adjustable roll is supported by two pivoted bearing holders whose positions are independently regulated by screw adjustment rods. As the side plates and bearing holders for the roll units are matched or jig-bored, no vertical tramming of the rolls is necessary. Once rolls

[2] Scale drawings are available from the Engineering Research Service, Research Branch, Dept. of Agriculture, Central Experimental Farm, Ottawa.

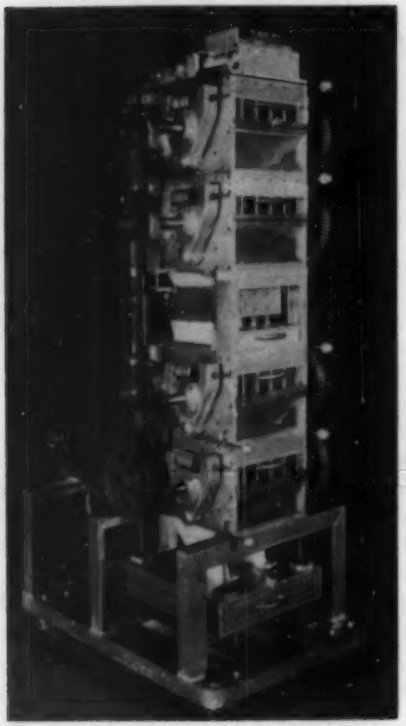

Fig. 1. Ottawa Micro Flour Mill without guards on sides.

are in position, further clearance settings may be made by a set-screw stop on a throw-out mechanism which moves both adjustable bearing holders simultaneously.

The first pair of break rolls are cut Allis Sharp 20 corrugations per in. and with a 1-in. spiral per 12 in. The rolls are operated dull to sharp with a differential of 2:1, a high-speed roll traveling at 500 r.p.m. The second pair of rolls are identical except they are cut 24 corrugations per in. The two pairs of reduction rolls are mat-finished and are operated with a differential of 2:1, the high-speed roll being operated at 500 r.p.m.

The bran bolter is located below the second break rolls. The stock is channeled to and from the bran bolter by nylon stockings. The separation of the four samples is maintained through the bran bolter. The coarser bran particles are removed by a 24 by 24 wire mesh sieve (aperture 0.0342 in.) which is vibrated $\frac{1}{8}$ in. at a rate of

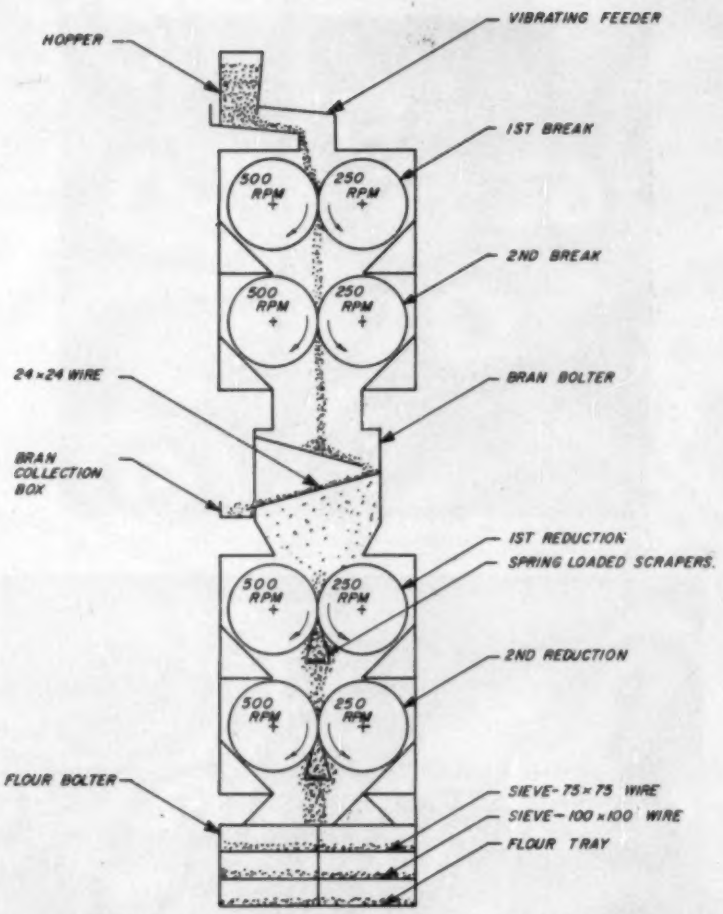

Fig. 2. Diagrammatic sketch showing operational principles.

1,500 cycles per minute. The bran removed from each sample is collected in a tray having four separate compartments.

The material which passes through the bran sieve continues to the reduction rolls. Spring-loaded plastic scrapers prevent any build-up of flour on the reduction rolls. Doors are provided front and back of each roll unit to facilitate cleaning after the stock has passed through the rolls. Brushing and low-pressure air may be employed for this operation.

Nylon stockings conduct the finely milled stock into a four-sec-

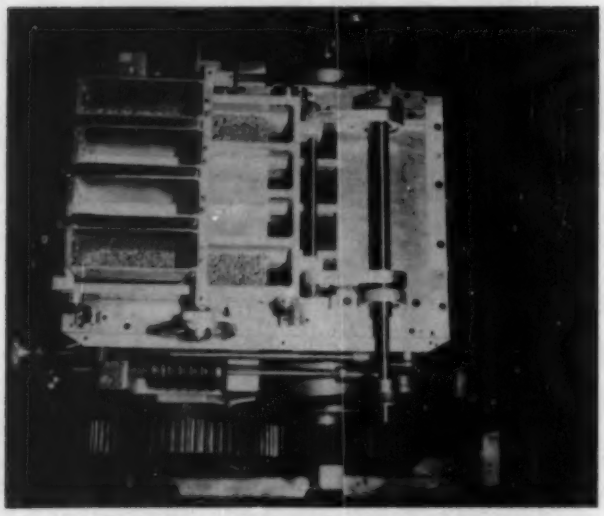

Fig. 3. The vibrator feeder arrangement at top of mill.

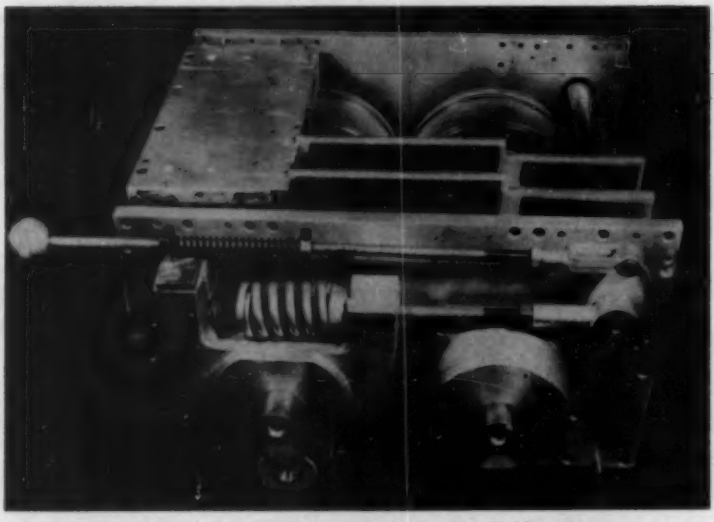

Fig. 4. Partially assembled roll unit showing divider plates.

tioned flour bolter. Final separation is accomplished by stainless-steel wire mesh sieves, 75 by 75 (aperture 0.0096 in.) and 100 by 100 (aperture 0.0055 in.). The flour bolter operates as a plansifter at 375

r.p.m. with a throw of ¼ in. Small flattened chains attached to the sieve frames, which are free to sweep the face of the sieves, accelerate the bolting action. Flour which passes through the 100 by 100 sieve is collected in four individual collection trays. The sieves and the flour collection trays pull out as one unit. Duplicate sieve and tray units may be used to increase milling capacity.

Preliminary Trials

In establishing a procedure for operating the mill, several trial runs were made with samples of hard red spring, hard red winter, soft white spring, and soft winter wheat. The hard wheat samples were tempered to 15.5% moisture before milling and the soft wheats to 14% by adding sufficient water the previous day. Except where noted, 102 g. of tempered wheat were milled. The rate of the feed was adjusted to deliver the samples to the first break rolls in 3 to 4 minutes. The bran yields recorded in Tables I, II, and III are the overs from a 30 by 30 wire mesh sieve. This sieve was changed to a 24 by 24 wire mesh for the later trials. The yields of shorts recorded in Tables I to IV are the overs from the 75 by 75 and the 100 by 100 wire mesh sieves. The flour is the product bolted through the 100 by 100 wire mesh sieve which has an aperture width between that of a 9xx and 10xx silk bolting cloth.

TABLE I

YIELD OF PRODUCTS FROM 102 GRAMS OF TEMPERED WHEAT FOR A 7-MINUTE MILL-RUN
(Six replications each roll position)

Roll Position	Bran[a]	Shorts	Flour	Total Products	Loss	Flour Moisture	Flour Ash
	g	g	g	g	g	%	%
1	22.1	14.2	60.9 ± 0.3	97.2	4.8	13.7	0.46
2	22.1	14.4	62.5 ± 0.3	99.0	3.0	13.8	0.47
3	22.1	13.4	62.1 ± 0.3	97.6	4.4	13.7	0.46
4	22.1	14.2	61.2 ± 0.3	97.4	4.5	13.7	0.47

[a] Overs on a 30 by 30 wire-mesh sieve.

The first trials were with a plump sample of hard red spring wheat. Six millings were made and the products from a 7-minute mill-run time are recorded in Table I. The yields of bran, shorts, and total products as well as flour moisture and flour ash are consistent for all roll positions and thus give confidence that comparable milling data can be obtained regardless of roll position. Mill loss per 102 g. of tempered wheat is low, since part of this loss may be attributed to moisture loss.

A second experiment was designed to determine the possibility of milling less than 100 g. of wheat. Drawing from the same sample referred to above, 100-, 75-, 50-, and 25-g. samples of tempered wheat were milled, and the data are recorded in Table II. The running time of the mill was 7 minutes in each case, as all four sample sizes were milled at the same time for each replicate.

TABLE II

YIELD OF PRODUCTS FROM 100, 75, 50, AND 25 G. OF TEMPERED WHEAT WITH 7-MINUTE MILL-RUN SCHEDULE

(Four replications each sample size)

SAMPLE SIZE	BRAN[a]		SHORTS		FLOUR		TOTAL PRODUCTS		LOSS		FLOUR ASH
	1[b]	2[c]	1	2	1	2	1	2	1	2	
g											%
100	23.0	23.0	14.1	14.1	59.3	59.3 ±0.6	96.4	96.4	3.6	3.6	0.46
75	17.2	22.9	8.4	11.2	45.8	61.1 ±0.6	71.4	95.2	3.6	4.8	0.46
50	11.2	22.4	4.4	8.8	31.1	62.8 ±1.0	46.7	93.2	3.3	6.6	0.46
25	5.0	20.0	1.1	4.4	15.3	61.1 ±1.8	21.4	85.6	3.6	14.4	0.50

[a] Overs on a 30 by 30 wire-mesh sieve.
[b] Actual yield in g.
[c] Calculated yield per 100 g. tempered wheat.

Variance analysis indicated no significant differences between roll position, replication, and calculated flour yield. The calculated flour yield per 100-g. sample was fairly uniform for all sample sizes in spite of a progressive increase in the calculated loss of total products as sample size decreased. It will be noted, however, that the calculated yield of shorts decreased with sample size, which would tend to offset a progressive decrease in the calculated flour yields. This would indicate that the clean-up on the shorts was more efficient with the smaller samples in the 7-minute mill-run and that the flour yields would have been progressively higher if the losses in total products were in proportion to the size of sample. One may conclude as well that an increase in flour yield would result from longer mill-run times for the larger samples. This led to the adoption of 10- and 13-minute mill-run schedules for the later trials. The good recovery of flour with sample size as low as 25 g. is worth noting.

A third experiment was set up in which samples of four varieties of hard red spring wheat were milled in a complicated test in which two variables were introduced. Two adjustments of the roll clearance in the second reduction unit were made, namely: 0.001 in. and <0.001 in. Speed ratios at 2:1 were maintained on the break roll units, but varied from 2:1 to 1¼:1 on the first and second reduction units. For each change, duplicate millings of the four samples were made, making sixty-four millings in all. Since the results indicated that

varying the speed ratios on the reduction rolls and the roll clearances in the second reduction unit made little difference in the flour yields, the data are presented in Table III only for the 0.001-in. clearance and for the 2:1 speed ratios for the four roll units. The roll clearances were 0.007 in., 0.003 in., 0.002 in., and 0.001 in. for the

TABLE III

VARIETAL DIFFERENCES IN MILLING BEHAVIOR
(Two replications each variety)

VARIETY	BRAN[a]	SHORTS	FLOUR	TOTAL PRODUCTS	FLOUR MOISTURE	FLOUR ASH	FLOUR[b]
	g	g	g	g	%	%	%
A	37.6	7.4	55.3	100.2	13.8	0.45	62.6
B	24.0	7.5	67.3	98.7	14.0	0.46	70.7
C	22.1	6.7	67.8	96.5	13.5	0.51	72.7
D	30.4	7.1	60.5	98.0	13.8	0.48	65.0

[a] Overs on a 30 by 30 wire-mesh sieve.
[b] Milled in a Buhler laboratory mill; flour obtained in % of 1,000-g. samples.

first break, second break, first reduction, and second reduction units respectively. The four samples were tempered to 15.5%, and 102 g. of tempered wheat were milled. The mill had a running time of 13 minutes, since two of the samples did not mill as freely as the other two.

The bran yields were lowest for samples B and C and highest for A and D. The lower flour yields for samples A and D reflected the higher bran yields for these two varieties. Corresponding wheats milled in a Buhler laboratory mill showed the same relationship in flour yields, thus indicating that the Ottawa micro mill gave similar information to the Buhler mill on the differences in the milling characteristics of these varieties.

A fourth experiment was conducted in which a hard red spring wheat sample and a soft white winter wheat sample were given 13-minute, 10-minute, and 7-minute mill-run times. The data are presented in Table IV.

The shorter mill-run schedules gave progressively higher yields of shorts at the expense of flour recovery for both samples. In addition the bran yields from the hard spring wheat sample were lower than from the soft winter wheat sample, thus contributing to lower flour recovery from the latter. The 7-minute mill-run time for the soft wheat sample is too short, as considerable flour was left unbolted on the 100 by 100 wire mesh sieve.

Four groups of samples representing different varieties and types grown in comparative test plots within each group were milled,

TABLE IV

MEAN MILLING YIELDS IN GRAMS FOR A HARD RED SPRING WHEAT SAMPLE AND A SOFT WHITE WINTER WHEAT WITH VARYING MILL-RUN SCHEDULES
(Eight replications each group)

MILL-RUN TIME	BRAN[a]	SHORTS	FLOUR	TOTAL PRODUCTS	LOSS
minutes					
Hard red spring wheat					
13	19.5	10.9	69.5 ±0.2	99.8	2.2
10	19.3	12.4	67.4 ±0.3	98.9	3.1
7	19.4	15.1	63.0 ±0.3	97.4	4.6
Soft white winter wheat					
13	23.5	6.9	65.1 ±0.4	95.3	6.7
10	24.1	10.2	63.2 ±0.3	97.4	4.6
7	24.8	21.5	51.8 ±1.9	98.3	3.7

[a] Overs on a 24 by 24 wire-mesh sieve.

TABLE V

FLOUR YIELDS IN GRAMS FOR DIFFERENT GROUPS OF WHEAT SAMPLES FROM 10- AND 13-MINUTE MILL-RUN SCHEDULES AND FROM A BUHLER LABORATORY MILL

GROUP	10-MINUTE	13-MINUTE	BUHLER-MILLED
Hard red spring			
A — 25 varieties	66.8	66.2	70.8[a]
B — 19 varieties	66.9	69.1	72.4[a]
Hard red winter			
C — 18 varieties	67.0	67.0	72.5[a]
Soft white spring			
D — 12 varieties	49.9	59.7	68.2[b]
Soft winter			
E — 8 varieties	55.4	65.9	71.2[a]

[a] Flour obtained in % of 2,000-g. sample.
[b] Flour obtained in % of 1,000-g. sample.

using 10-minute and 13-minute mill-run schedules. The mean flour yields are recorded in Table V along with the corresponding mean yields for the same wheat samples milled in a Buhler laboratory mill.

Groups A, B, and C gave almost the same mean flour yields for both mill-run schedules, but the softer types in D and E required the longer time for the higher flour yields. None of the yields was as high as that obtained with the Buhler laboratory mill, a reflection of the effect of the shorter system of the Ottawa micro flour mill.

Some indication of the mill capacity at 10- and 13-minute mill-run schedules was obtained with one series of spring wheat samples. Twenty-four samples were milled in 78 minutes with a 10-minute schedule and 24 samples in 98 minutes with a 13-minute schedule. The calculated rates are 18.4 samples per hour for the former and 14.6 samples per hour for the latter. Only the flour yields were recorded in these trials.

Discussion

The Ottawa micro flour mill was designed to mill small samples of wheat for quality tests on the flours. This is accomplished through the use of a short system requiring a minimum of manual labor and allowing for the recovery of a substantial proportion of the available white flour. The two-break system gives lower yields of bran for hard wheat than for soft wheat, and thus should be valuable in differentiating between hard and soft types. The mill should assist as well in distinguishing between free and less free milling wheats within the same class on the basis of flour yield. Since mill-run time for best results is a variable the choice of schedule will depend on the milling behavior of the samples. Sufficient time should be given in order that a good clean-up of the stock on the 100 by 100 wire-mesh sieve is obtained for the reference samples.

The principle of milling four samples at one time enables the operator to mill a substantial number of samples in a short time. While the mill is designed to handle individual samples of 100 g. or less, up to 400-g. samples may be milled at one time in increments of 100 g. each. Low mill losses and the easily accessible features for cleaning the mill are an asset for handling small samples.

The mill should be useful in wheat breeding programs, not only in assessing milling behavior but through subsequent tests on the flour produced by it. It should assist as well in genetic studies of quality components where large numbers of samples are required to be milled.

Acknowledgments

The authors are indebted to W. Kalbfleisch, to R. H. Cunningham, and to others who have taken an interest and have contributed to the development of the mill.

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2(3)-BENZOXAZOLONE IN MALTING OF BARLEY[1]

MATTI LINKO, PEKKA LINKO, AND TOR-MAGNUS ENARI[2]

ABSTRACT

Experimental malting of barley showed that 2(3)-benzoxazolone (BOA), alone and together with a small quantity of gibberellin, increased malt yield by decreasing rootlet growth and respiration. Extract content and modification were improved. The activities of alpha- and beta-amylase remained unaffected or were somewhat increased. Best results were obtained using BOA together with gibberellin; with a short germination time it was possible to obtain malt of a high extract content and alpha-amylase activity. Furthermore, malt yield was increased by about 2%.

Several natural and synthetic compounds have been found to inhibit root growth during germination, e.g., indole-3-acetic acid (9), alpha-naphthylacetic acid (3), coumarin (6), and 2,4-dichlorophenoxyacetic acid (7). Kirsop and Pollock (5) have recently shown that the embryo can be removed after the first 3 days of germination without affecting properties of the resulting malt other than beta-amylase activity, which is decreased. These findings have given theoretical background for the use of root growth-restricting substances in malting. Plant-growth regulators of another type are gibberellins, which increase the speed of modification. Thus the time of germination can be reduced, and yet malt of better quality is obtained (11,12). Gibberellins can advantageously be used together with substances restricting root growth (7).

In a series of preliminary germination tests, 2(3)-benzoxazolone (BOA) gave some promising results in the restriction of rootlet growth (8). A further advantage of this compound is that mold growth is prevented or delayed. By proper treatment alpha-amylase and protease activities were not significantly lowered. However, in these preliminary studies malting conditions differed considerably from those of the technical process, and the samples were too small for a complete analysis of the final malt.

In the present study some further malting experiments were performed with BOA and gibberellin using a pneumatic-type experimental malting apparatus.

Materials and Methods

2(3)-Benzoxazolone was synthesized according to Bywater *et al.*

[1] Manuscript received April 28, 1960.

[2] Respectively: Chemist, Laboratory of Brewing, Helsinki, Finland; Assistant Professor, Kansas State University, Manhattan; and Director, Laboratory of Brewing, Helsinki, Finland.

(2). The barley varieties used were Pirkka, a Finnish 6-row variety, and Balder, a Swedish 2-row variety. The experimental malting apparatus had a capacity of 8×1 kg. of barley. The apparatus consisted of a steeping tank, a pneumatic-type germination chamber, and a kiln. The steeping tank was equipped with a pump for water circulation and with a small compressor for aeration. The temperature of steeping water could be controlled. The germination chamber was equipped for circulation of moist, temperature-controlled air through the grains. The quantities of fresh and recirculating air could be controlled with perforated plates. In the kiln, heated air was forced through the grains, and the temperature was automatically controlled and registered. A detailed description of the apparatus will be published elsewhere[3]. The following malting process was employed:

Steeping. Four different methods of steeping were used:

a. Normal steeping involving spraying with a solution of BOA after 31 hours. The grains were first steeped at 11°–12°C. for 23 hours in water, which was changed at intervals of 1, 6, and 16 hours. This was followed by dry-steeping for 8 hours. Then the grains were sprayed with 10 ml. of a solution of BOA (448 to 672 mg. BOA per 10 ml., corresponding to 500 to 750 mg. BOA per kg. barley, d.m.) in 80% ethanol (the control samples were sprayed with 80% ethanol). The process was then continued by dry-steeping only. The grains were sprayed with water at about 6-hour intervals to increase the moisture content. After a total steeping time of 55 hours the moisture content of the barley was 43%. Aeration was continuous during the whole process.

b. Normal steeping involving standing in a water solution of BOA at the end of the process. The grains were first steeped at 11°–12°C. for 20 hours in water, which was changed at intervals of 2, 4, 6, and 8 hours. After this followed dry-steeping for 9 hours, again steeping in water for 0.5 hour, dry-steeping for 6.5 hours, steeping in water for 0.5 hour, and dry-steeping for 8.5 hours. Finally the grains were allowed to stand for 2 hours in a 0.05% water solution of BOA (or water in the control sample), followed by dry-steeping for 8 hours. Thus the total steeping time was 55 hours. The final moisture content of barley was 45%. The aeration was continuous, except for the standing in BOA-solution.

c. Steeping without aeration. In this process there was no aeration during the steeping in water. The grains were first allowed to stand

[3] Linko, M. Experimental maltings in the Laboratory of Brewing, Helsinki. *Mallasjuomat* (to be published).

for 20 hours in a 0.05% water solution of BOA (or water in the control sample). After this followed dry-steeping with aeration for 9 hours, standing in BOA-solution for 7 hours, and finally dry-steeping for 9 hours. The total steeping time was 45 hours, temperature about 14°C., and the final moisture content of barley 44%.

d. Steeping by spraying. In this process the grains were first steeped in water for 2 hours, but after this the moisture content was increased only by spraying with 0.05% water solution of BOA at 6-hour intervals (the control sample was sprayed with water). The total steeping time was 60 hours, temperature 11°–12°C., aeration continuous, and the final moisture content of barley 43%.

Germination. The steeped barley was germinated for 5 to 7 days. The initial temperature was 12°C. In the beginning the temperature was raised by 1°C. per day, until 16°C. was reached. This temperature was then maintained through the rest of the germination period. Moisture content was adjusted to 43 to 44%, and the grains were turned by hand once a day.

Kilning. The temperature was first raised to 52°C. within 1 hour and maintained there for 3 hours. This was followed by a raise to 60°C. during 5 hours, which temperature was again maintained for 4 hours. Finally temperature was increased to 86°C. during 5 hours, and maintained there for 3 hours; total kilning time, 21 hours.

The rootlets were removed after kilning by brushing against a steel wire net, and resulting malts and rootlets were weighed.

Analysis of malt. The resulting malts were analyzed according to the standard methods of the European Brewery Convention (4), which do not essentially differ from those of the American Society of Brewing Chemists. Alpha-amylase activity was determined according to the ASBC method (1), and beta-amylase activity was calculated from the values of total diastatic power and the diastatic effect of alpha-amylase. This calculation was based upon the additivity of the diastatic effects of alpha- and beta-amylase (10).

The mashings of finely and coarsely ground malts and the respective determinations of extract content, as well as the determinations of moisture content, total nitrogen, Kolbach index, and acrospire length were performed in duplicate. According to earlier experience based on replicate maltings, differences exceeding 0.3% in the values of extract content, 0.5% in malt yield, and 0.3% in the loss by rootlets or by respiration can be considered as significant.

Results and Discussion

The results from the first series of maltings made with Pirkka bar-

TABLE I

THE EFFECT OF 2(3)-BENZOXAZOLONE (BOA) AND GIBBERELLIN ON MALT QUALITY AND MALT YIELD OF BARLEY VARIETY PIRKKA

(Steeping Procedure *(a)*; 500 to 750 p.p.m. BOA added as an alcohol spray after 31 hours of steeping; 0.5 p.p.m. gibberellin added as a water spray after 33 hours of steeping; germination time 7 days)

BOA Added (p.p.m.):	0	750	0	500
Gibberellin Added (p.p.m.):	0	0	0.5	0.5
Moisture, %	3.7	3.6	3.5	3.6
Extract, fine grind (d.m.), %	80.6	80.7	81.9	82.1
Extract, coarse grind (d.m.), %	76.4	76.4	78.9	79.5
Difference fine-coarse (d.m.), %	4.2	4.3	3.0	2.6
pH of the laboratory wort	5.75	5.70	5.65	5.70
Saccharification time, minutes	<10	<10	<10	<10
Clarity of the laboratory wort	clear	clear	clear	clear
Speed of filtration	normal	normal	normal	normal
Total nitrogen (d.m.), %	2.08	2.09	2.06	2.08
Kolbach index, %	59	61	67	69
Acrospire length: 0–¼	5	6	3	3
¼–½	4	4	7	6
½–¾	58	66	17	23
¾–1	33	24	64	56
overgrown	0	0	9	12
Average acrospire length	0.68	0.66	0.82	0.81
Malt yield (d.m.), %	89.1	90.1	89.4	89.6
Loss by rootlets (d.m.), %	4.1	3.6	3.8	3.5
Loss by respiration and steeping (d.m.), %	6.8	6.3	6.8	6.9
Malt yield × extract yield (d.m.), %	71.8	72.7	73.2	73.6

ley according to the steeping procedure *a* are presented in Table I. The results show that the quality of malt was not affected by BOA. Malt yield was increased by 1% owing to a 0.5% decrease in the growth of rootlets and to a 0.5% retardation in respiration. The acrospire growth was not affected, or was slightly reduced. The addition of gibberellin[4] caused a marked increase in the extract content, in the acrospire growth, and in the speed of modification, as could be expected, but the growth of rootlets was not increased. In these samples malt yield was almost unaffected by BOA, perhaps owing to the fact that the samples were overgrown. However, the modification was somewhat improved.

The next series of maltings was made with Balder barley according to steeping procedures *b*, *c*, and *d*. In two cases a small amount of gibberellin (1 p.p.m.) was added as water spray during the first day of germination. In these two samples germination time was shortened by 2 days. The amount of BOA in the grains was not determined, but according to earlier studies concerning BOA uptake it can be assumed to be of the order of 200 to 300 p.p.m. when steeping

[4] Purum grade, Fluka AG, Chemische Fabrik, Buchs SG, Switzerland.

TABLE II

EFFECT OF 2(3)-BENZOXAZOLONE (BOA) AND GIBBERELLIN ON MALT QUALITY AND MALT YIELD OF BARLEY VARIETY BALDER

BOA Added	−	+	+	−	+	−	+	+
Gibberellin Added (p.p.m.)	0	0	1	0	0	0	0	1
Steeping Procedure (See Text)	*b*	*b*	*b*	*c*	*c*	*d*	*d*	*d*
Germination Time (days)	7	7	5	7	7	7	7	5
Moisture, %	3.7	3.5	3.6	3.6	3.6	3.6	3.6	3.6
Extract, fine grind (d.m.), %	78.6	79.1	80.7	79.0	79.7	78.6	78.5	80.0
Extract, coarse grind (d.m.), %	75.4	76.4	77.3	75.8	77.2	74.4	74.5	76.4
Difference fine-coarse (d.m.), %	3.2	2.7	3.4	3.2	2.5	4.2	4.0	3.6
pH of the laboratory wort	5.85	5.95	5.80	5.90	5.95	6.00	6.05	6.00
Saccharification time, minutes	>10	>10	>10	>10	>10	10–15	>10	>10
Color, °EBC	3.1	3.4	5.3	2.7	4.1	3.4	3.4	4.1
Clarity of the laboratory wort	clear	clear	clear	clear	clear	clear	clear	clear
Speed of filtration	normal	normal	normal	normal	normal	normal	normal	normal
Total nitrogen (d.m.), %	1.77	1.76	1.79	1.74	1.79	1.76	1.77	1.74
Kolbach index, %	45	47	57	44	49	43	42	53
Alpha-amylase, 20° D.U.	34	37	44	33	35	28	32	40
Beta-amylase, W.K.	240	250	240	210	210	210	220	200
Acrospire length, %: 0–¼	1	2	1	1	4	2	2	2
¼–½	2	1	3	2	4	8	2	9
½–¾	84	86	77	86	85	63	83	59
¾–1	13	11	16	11	7	27	11	29
overgrown	0	0	3	0	0	0	2	1
Average acrospire length	0.65	0.65	0.67	0.65	0.62	0.67	0.67	0.67
Malt yield, %	88.7	89.7	92.1	89.6	91.6	89.8	90.5	92.7
Loss by rootlets (d.m.) %	5.2	4.3	3.2	4.6	3.1	4.7	4.2	3.2
Loss by respiration and steeping (d.m.), %	6.1	6.0	4.7	5.8	5.3	5.5	5.3	4.1
Malt yield × extract yield (d.m.), %	69.7	71.0	74.3	70.8	73.0	70.6	71.0	74.2

procedures *c* and *d* were used, and about 20 p.p.m. when the steeping procedure *b* is used. The results are presented in Table II.

The most striking results were obtained with steeping procedure *c*, without aeration during the water steep. In these samples the growth of rootlets began very slowly as compared with other samples steeped with efficient aeration. No rootlets could be seen after the first day of germination in grains steeped in BOA-solution (Fig. 1).

Fig. 1. The effect of 2(3)-benzoxazolone (BOA) on the growth of rootlets during malting of barley, variety Balder. Steeping procedure *b* (see text for details). Top left: control sample after first day of germination; top right: control sample after second day of germination; bottom left: sample steeped in BOA after first day of germination; bottom right: sample steeped in BOA after second day of germination.

However, once started, growth continued almost normally. In these maltings BOA caused a 2% increase in the malt yield. The loss by rootlets was decreased by 1.5% and the respiration loss by 0.5%. Furthermore, the quality of malt was improved; the extract content was increased by 0.7%, and the modification was better as judged from the smaller fine/coarse grind extract difference and the increased Kolbach index (wort soluble N as percent of total N). The increase in the extract content results, no doubt, at least partly from the retardation of respiration, although manometric techniques with a fairly short measuring time failed to detect any decrease in carbon di-

oxide production (8). The lack of aeration did not impair the quality of malt, as can be seen by comparing the control sample with other control samples.

In the samples steeped only by spraying (procedure *d*), the effect of BOA was small as compared to the samples steeped according to the procedure *c*, although the BOA uptake must have been of the same order. This seems to be associated with the more aerobic conditions, which might cause metabolizing of BOA.

In the samples steeped according to the procedure *b* the effect of BOA was stronger than could be expected. These samples were standing in BOA-solution only for 2 hours, and the uptake of BOA was respectively less than one-tenth as compared with the other samples. Despite this, there was an increase of 1% in malt yield and 0.5% in the extract content. The fine/coarse grind extract difference was decreased by 0.5%, showing a better modification.

In all cases acrospire growth remained unaffected or was slightly reduced by BOA. Respiration was somewhat retarded, and the activities of alpha- and beta-amylase slightly increased or maintained at the same level as in the control samples. In these respects the effects of BOA are different from those of 2,4-D, which stimulates acrospire growth, does not affect respiration, and decreases alpha-amylase activity (7).

The best results were obtained by using BOA together with a small quantity of gibberellin (1 p.p.m.). Owing to the fact that germination time could be reduced by 2 days, malt yield was increased appreciably (about 3%). Furthermore, the extract content of the resulting malt was materially higher (1.5 to 2.1%). In spite of the shortened germination time, alpha-amylase activity increased about 30%. Proteolytic activity was also high as judged from the Kolbach index, but beta-amylase activity was not significantly changed.

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MEASUREMENTS OF THE PARTICLE SIZE DISTRIBUTION OF FLOUR[1]

R. R. IRANI[2] AND W. S. FONG[3]

ABSTRACT

The particle size distribution of flour, measured microscopically by gravitational sedimentation, centrifugal sedimentation, and sieving, all gave mean diameters and distribution curves which agreed with one another within experimental error. Measurements using changes in electrolytic resistivity gave particle size distributions that deviated significantly and nonrandomly from the other methods.

The effect of particle size distribution on the quality of flour, including the ratio of starch to protein content, has been known for many years, and studies are still being carried on (8). Recently, interest in particle size distribution measurements has mushroomed, and several new techniques and commercial instruments have been developed.

Sieving, sedimentation, and photoextinction techniques have received most of the attention in the measuring of particle size distribution of flour. Whitby (24,26) and Heywood (12) discussed the mechanics of fine sieving and concluded that sieving can be divided into two different steps. During the first step, particles with a size

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much smaller than the sieve opening pass through. During the second and relatively slower step, particles whose size is close to that of the opening are sieved through.

Kent-Jones *et al.* (18) describe a simple sedimentation procedure for determining flour granularity. Heald (9) used a mixture of carbon tetrachloride and naphtha as the sedimentation fluid and utilized an adaptation of the Andreasen method (3) to measure the particle size distribution of flour.

The photoextinction technique has received much attention. Kent-Jones *et al.* (18,19) described a photosedimentation method in which the percentage of different-sized particles in flour is correlated with the decrease in cloudiness with time. Amos (2), using this photosedimentation method, concluded that small changes in the granularity of flour do not affect its quality for biscuit production. All known photoextinction methods, whether sedimentation is involved or not, are questionable because of the large errors (21) involved due to dependence of the extinction coefficient on particle size and shape factors (20). For example, Deschreider (7) found that the turbidity of flour suspensions rose with decreasing particle size and increasing ash content even when the flour concentration in the suspension was kept constant.

Hildebrand *et al.* (11) discussed the advantages and disadvantages of microscopic determinations of particle size of flour; they also concluded that sieving and sedimentation become inaccurate below 200 mesh (74μ) and above 60μ respectively. On the other hand, Wichser and Shellenberger (27) compared Ro-Tap sieving with elutriation and sedimentation (Andreasen) and concluded that sieving was most applicable above 37μ. Whitby (23) discussed various methods of measuring the particle size distribution of flour and pointed out that some of the inconsistencies are probably due to differences in particle shape and density.

In discussing particle size distributions, it is essential to define "size" for a particle with an irregular shape. If the center of gravity of an irregular particle is located, an extremely large number of lines terminating at the edges of the particle but passing through the center can be drawn. These lines are diameters of the particle. The average of these diameters, either arithmetic or geometric, will be used as the definition of particle size[4] throughout this paper.

This paper will present a comparison of various methods of measuring particle size distribution of soft wheat flour. The methods

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comprise microscopy, gravitational sedimentation, centrifugal sedimentation, sieving, and changes in the electrolytic resistivity.

Methods that give only an average size and no distribution will not be considered, because in most cases these so-called averages do not adequately represent the entire size distribution and are often affected by properties of the powder other than size, e.g., permeability measurements (13).

Materials and Methods

Materials. Ten commercially milled soft wheat flours were employed. A commercial grade of tricalcium phosphate, having a geometric mean weight size of 2.3μ (98% between 0.3 and 7μ) with a calcium oxide to phosphorus pentoxide molar ratio of 3.18 and exhibiting an apatite X-ray pattern (22), was added to the flour when dry sieving was utilized.

Gravitational Sedimentation. The sedimentation measurements were made with a Monsanto-automatized Bostock type (5) liquid sedimentation balance, previously described (1). The dispersions were obtained by the gradual dilution of the weighed sample of flour, and examined microscopically to verify the deagglomeration of the particles. The volume fraction of the powder in the dispersion was always kept below 0.3% to ensure free settling.

The liquid sedimentation data for an initially homogeneous suspension were interpreted to give particle size distributions by the methods outlined by Oden and modified by Bostock (5):

$$w = W - \frac{dW}{d\ln t} \tag{1}$$

where W is the settled weight at time, t, and w is the weight of that fraction of settled particles whose size, M, would allow sedimentation through the entire height of the column, h, as given by Stokes' equation

$$M = \sqrt{\frac{18\,\eta\,h}{(\rho_1 - \rho_2)\;g t}} \tag{2}$$

where η and ρ_2 are the viscosity and density of the sedimentation fluid, respectively, and g the acceleration due to gravity .The particle density, ρ_1, was measured pycnometrically (6).

Flour is known to be a heterogeneous mixture of particles of different densities and shapes. Duplicate specific gravity determinations of five flour samples and their air-classified fractions gave values ranging from 1.40 to 1.50 with an average value of 1.44. The

average value was used throughout the calculations, causing an expected maximum error of 5% in the calculated M. The effect of shape factors on equation 2 will be discussed later.

Centrifugal Sedimentation. The apparatus was a commercial unit[5], previously described (25), that employs capillaries as a means of collecting and measuring the amount of material during sedimentation. The sedimentation fluids were either benzene or ethanol. One of the assumptions in this method is that the sediment height is proportional to the weight settled. This was found to be true for many powders including flour, provided gravitational and centrifugal sedimentation were not combined, or centrifuges of different r.p.m. were not interchanged during a determination; otherwise, a bulk density correction factor was evaluated and used as follows.

First, all particles in the sedimentation tube were allowed to settle freely under gravity and the height, h_1, was recorded. The same tube was then centrifuged for 30 minutes each at 600, 1,200, and 1,800 r.p.m., and the heights, h_2, h_3, and h_4, were observed. Since during a size determination the final height was read after centrifugation at 1,800 r.p.m., the heights obtained with no centrifugation, with centrifugation at 600 r.p.m., and with centrifugation at 1,200 r.p.m. were multiplied by h_4/h_1, h_4/h_2, and h_4/h_3 respectively. The corrections were also found to vary slightly from one sedimentation fluid to another.

Microscopic Counting. The microscopic sizing and counting measurements were made electronically with a Flying-Spot Particle Resolver[6] according to previously described procedures (1). The resulting number-size distributions were converted to weight-size distributions as follows:

$$\frac{y}{100} = 1 - \frac{\sum_{M_1=0}^{M_1=M} f_i M_i^3}{\sum_{M_1=0}^{M_1=\infty} f_i M_i^3} \qquad (3)$$

where y is the percent by weight greater than the size, M, and f_i is the frequency of occurrence by number of the size, M_i. In equation 3 it was assumed that the particle density is independent of size, and the summation terms were evaluated by performing trapezoidal integration on the histogram.

[5] Manufactured by Mine Safety Appliance Co., Pittsburgh, Pa.
[6] Manufactured by Cinema Television Company, Ltd., London, England.

Sieving. Micromesh sieves[7], calibrated according to a previously described procedure (16), were utilized. The flour samples were sieved either by brushing or with a Ro-Tap shaker. In the latter technique, it was found that the addition of 1% conditioner grade tricalcium phosphate increased the rate of sieving several-fold and minimized sieve blinding through improving the flow properties of the flour (17).

Electrolytic Resistivity Changes. The particle size distribution measurements involving changes in electrolytic resistivity were made according to the procedure described by Berg (4), utilizing a commercial unit[8]. The instrument calibration was obtained for each orifice opening using extremely uniform Ragweed Pollen having a size of 18.6μ, determined microscopically from measurement of hundreds of particles. The electrolyte was a 4% solution of ammonium potassium thiocyanate in isopropanol.

Results and Discussion

Duplicate measurements or more were made on all of the investigated methods. The precision of the evaluated techniques was found to be at least as good as previously reported. Thus, the precision of measuring geometric mean sizes of the particle size distribution was ±5% * for gravitational sedimentation, ±6% * for centrifugal sedimentation, ±3% * for micromesh sieving, ±5% * for microscopy, and ±4% * for the Coulter counter. It is important to remember that these values represent *precision* and not *accuracy*. The precision found in this study was better than that previously reported for sedimentation and microscopy (1) because of improved training of operators.

The excellent agreement obtained, shown in Table I, in the particle size data when ethanol, benzene, isobutanol, or 2-ethyl hexanol was employed as the settling medium is evidence that proper deagglomeration was achieved. Analysis of the data listed in Table I indicated that the reproducibility of measuring the geometric mean size was better than ±5% *.

Heiss and Coull (10) investigated the effect of orientation and shape on the settling velocity of nonisometric particles in a viscous medium. Their data indicate that the fairly chubby flour particle is expected to settle with a velocity that is surprisingly close (within 6%) to that of a sphere of equal volume.

In sieving, microscopy, and sedimention the computed sizes as

[7] Manufactured by Buckbee Mears Co., St. Paul, Minn.
[8] Manufactured by Coulter Electronics, Inc., Chicago, Ill.

TABLE I

PARTICLE SIZE DISTRIBUTION BY WEIGHT FOR A SAMPLE OF FLOUR FROM SEDIMENTATION IN VARIOUS SOLVENTS

Size	Percent by Weight Greater than			
	Ethanol	Benzene	Isobutanol	2-Ethyl Hexanol
μ				
5	100	99.5	99.5	99.2
10	98.1	96.2	97.8	95.1
20	80.0	83.0	85.1	84.9
30	65.1	70.9	71.2	68.7
40	44.0	49.0	46.0	48.0
50	37.0	36.0	38.0	39.0
60	30.0	29.5	28.0	30.0
80	17.0	19.0	20.0	18.0
100	5.0	4.0	7.0	7.0
120	0.0	1.0	0.0	1.0

previously defined are some averages of the diameters of the particles. The arithmetic and geometric averages, known to give the largest difference between averages, have recently been shown (see footnote 4) to differ by only 6.0% for a particle whose largest diameter is three times the smallest diameter. This finding justifies the intercomparison of various methods for particle size distribution measurements of flour.

Even though ten flour samples were investigated, for the sake of brevity the results are presented on only two samples. The particle size distributions obtained by the various methods for two flours with geometric mean particle sizes of about 45 and 8μ are given in Tables II and III respectively.

The centrifugal sedimentation method was not used on the coarser sample because the lowest r.p.m. centrifuge caused substantial amounts of sediment to settle in a matter of seconds.

Only incomplete sieving data are presented on the finer flour sample because no sieves have been manufactured in the size range below 10μ.

For both samples of flour, all investigated methods agree with one another within experimental error except for the Coulter counter, which gave results systematically different from microscopy and sedimentation. This discrepancy has been discussed by Irani and shown to be resolvable (15) for flour provided the Coulter counter is calibrated with flour rather than with an arbitrary powder. In agreement with previous work (1) the data obtained by microscopy and sedimentation agree with one another in all cases.

In agreement with previous findings (16), the results indicate that the recently developed micromesh sieves, when calibrated, and in combination with a conditioning agent, namely, tricalcium phos-

TABLE II

INTERCOMPARISON OF METHODS ON FLOUR

Size	Percent by Weight Greater than by				
	Microscopy	Sieving[a]	Sieving[b]	Gravitational Sedimentation[c]	Coulter Counter
μ					
5	99.6	..	..	100.0	99
10	98.5	..	..	98.2	95
15	94.0	..	..	93.2	91
20	87.0	..	..	82.0	82
25	81.0	..	..	76.0	74
30	74.0	73	78	68.0	68
35	...	..	..	60.0	62
40	65.0	..	..	54.0	56
45	...	58	63	48.0	52
50	43.0	..	..	38.0	..
55	...	..	..	...	..
60	32.0	42	28	30.0	30
70	...	..	..	20.0	11
80	18.0	18	2	...	1
90	...	..	..	...	..
100	7.0	..	..	5	0
120	0	..	..	0	0

[a] Brushing.
[b] Ro-Tap with 1% tricalcium phosphate.
[c] Average of data obtained in ethanol, isobutanol, and 2-ethyl hexanol.

phate, are accurate down to 15μ, whereas conventional woven sieves become inaccurate below 50μ (1,11). Attempts to calibrate woven sieves below 50μ were unsuccessful.

Figure 1, the plot of the data on log-probability graph paper such as K & E 359-24, reveals that the finer flour sample investigated follows a log-normal distribution, whereas the coarser flour sample investigated represents an abnormal log-normal distribution that had been artificially scalped at 120μ (14).

TABLE III

INTERCOMPARISON OF METHODS ON FLOUR

Size	Percent by Weight Greater than by				
	Microscopy	Gravitational Sedimentation[a]	Centrifugal Sedimentation[b]	Sieving[c]	Coulter Counter
μ					
2	99.6	99.3	...	...	100.0
3	97.5	97.0	95	...	100.0
4	89.0	89.0	..	...	100.0
6	68.0	...	60	...	98.8
8	44.0	46.0	34	...	...
10	29.0	...	20	...	72.0
15	10.0	10.0	6	16.0	45.0
20	3.5	2.0	..	6.0	12.0
30	0.5	...	..	1.2	1.9

[a] Average of data obtained in ethanol, isobutanol, and 2-ethyl hexanol.
[b] Average of data obtained in ethanol and benzene.
[c] Brushing.

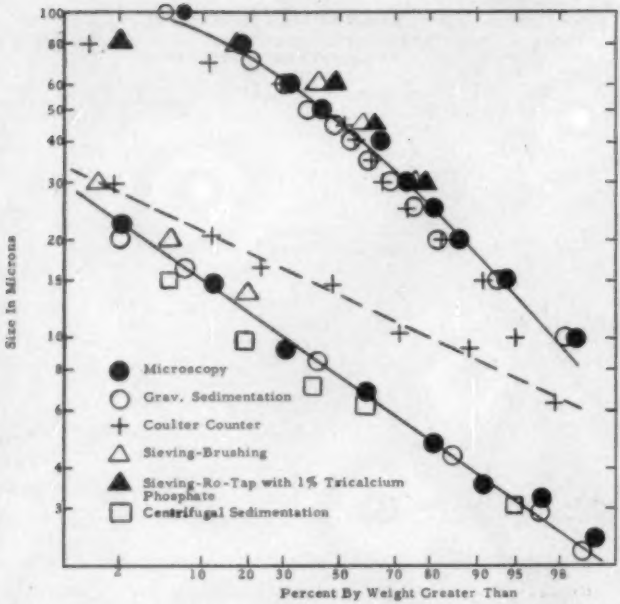

Fig. 1. Intercomparison of methods for measuring the particle size distribution of flour.

Many control tests on flour require a knowledge of the percent finer than or coarser than a *specific size*. When this percentage is less than 10% or larger than 90%, it is obvious, from Fig. 1, that the control limits must be set for each technique independently. While the various methods with calibration or otherwise agree with one another as far as the average size and the shape of the distribution curve are concerned, they give results that are significantly different from one another if only one point on the distribution curve is considered. Therefore, one-point limits, which are very inadequate in describing size distributions, should be set for each method independently. In addition, for these limits to be at all meaningful, they should not approach the precision of the specific technique.

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CHANGES IN THE CHEMICAL COMPOSITION AND IN THE DISTRIBUTION OF NITROGEN OF MAIZE AT DIFFERENT STAGES OF DEVELOPMENT[1]

RICARDO BRESSANI[2] AND RODOLFO CONDE[2]

ABSTRACT

On a moisture-free basis, the nitrogen, crude fiber, and ash contents decreased, while the ether extract and carbohydrate content increased with maturation of the corn kernel. The alcohol-soluble nitrogen (zein) increased with development of the grain, the alkali-soluble nitrogen increased at first but decreased 16 days after flowering. The acid-soluble nitrogen of the corn kernel constantly decreased with maturation. The concentration of eight amino acids, in terms of nitrogen of the kernel, was followed during grain maturation. The results indicated that those amino acids found in significant amounts in zein increased, while those found in relatively small amounts in this protein fraction decreased.

In the protein synthesis which accompanies the development of seeds, the amount of protein nitrogen increases at the expense of soluble nitrogen (15–17). Cereal grains are known to contain large amounts of alcohol-soluble protein (2,5,8,9,12,19), and in the mature corn kernel this protein fraction, zein, constitutes about 40 to 50% of the protein nitrogen of the grain (2,8,9,12,17). Several investigators have demonstrated that in the mature corn kernel, alcohol-soluble nitrogen correlates highly with total nitrogen. Furthermore, any changes taking place in the nitrogen of the corn, through breeding (4,6,18) or fertilization (8,18), are reflected in changes in the alcohol-soluble nitrogen of the seed. Except for the work of Zeleny (21), no report has appeared of the time at which the zein is formed in the maturing corn kernel. Zeleny (21) showed that zein is nearly absent in the very immature kernel, but is synthesized at a rapid rate as the grain approaches maturity. He also found that the rapid increase in the ratio of zein nitrogen to total nitrogen is almost exactly paralleled by the decrease in water-soluble nonprotein nitrogen. With the development of a new method of extraction and fractionation of corn proteins by Mertz *et al.* (11,12), it was of interest to study the changes taking place in some nitrogen compounds during the development of the grain.

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Materials and Methods

The seed samples were collected from an open-pollinated corn cultivated on a plot of about 400 sq. meters in the southern coast of Guatemala, at an elevation of 650 ft. The planting consisted of hills spaced 15 in. apart in rows spaced 42 in. apart; each hill contained from one to three plants. The corn was planted in early May 1956 and reached flowering stage in mid-June. Sample collections were begun on June 24 and continued every 7 days until August 18 for a total of nine samples. Every collection contained one ear of corn from each of 15 to 20 randomly selected hills. The samples were collected from 9 to 10 a.m., transported to the laboratory within about 3 hours, and stored at −20°C. Each ear of corn was shelled and the seeds were frozen and ground immediately in a Waring Blendor or Wiley mill. The ground samples were kept frozen at all times, except during the chemical analyses.

Proximate analyses of the grain were carried out as described by the methods of the AOAC (1), using the vacuum-oven method for moisture determinations. Nitrogen was determined in the vacuum oven-dried and wet samples by the Kjeldahl method, selenious acid being used as catalyst; distillation and titration were carried out according to the recommendations of Hamilton and Simpson (7). The method of Mertz *et al.* (11,12) was used for the extraction and fractionation of the nitrogen of the whole ground kernel. For the determination of free ammonia and free amino acids, subsamples of the ground kernel estimated to contain 20 to 30 mg. of dialyzable nitrogen were extracted for 24 hours at 4°C. with 60 ml. of sodium phosphate buffer (pH 6.8–7.0), centrifuged, and the supernatant was collected. The residue was suspended in 25 ml. of water and again centrifuged; the two supernatant solutions were then combined; their volume was measured and the nitrogen content determined by the micro-Kjeldahl method (1). To determine dialyzable nitrogen, 25 ml. of the phosphate buffer extract and of the acid-soluble fraction of the copper sulfate method (11,12) were placed in Cellophane tubing of 19-mm. diameter, surrounded by 50 ml. of the same buffer in the case of the phosphate buffer extract, and by 75 ml. of distilled water in the case of the acid-soluble fraction. After 40 hours at 4°C. the volume of the dialysate was measured and its nitrogen determined. Free ammonia was determined in the phosphate buffer extract by methods of Vickery and Meiss (20), and total free amino acids by the colorimetric method of Moore and Stein (13). Several amino acids were determined microbiologically by means of *Leuconostoc mesen-*

teroides and Difco[3] media.

Results

The changes taking place in the proximate chemical composition of the corn kernel during the development of the grain are shown in Table I. No major changes in moisture took place before 16 days

TABLE I

CHANGES IN THE CHEMICAL COMPOSITION OF THE DEVELOPING CORN KERNEL DETERMINED ON MOISTURE-FREE SAMPLES

Days after Flowering	Moisture	Nitrogen	Proximate Analysis, Dry Moisture Basis				
			Protein[a]	Ether Extract	Crude Fiber	Ash	Carbohydrates by Difference
	%	%	%	%	%	%	%
10	89.0	2.81	17.6	2.0	9.1	3.7	67.6
16	88.8	2.98	18.6	2.0	8.2	4.3	67.0
23	72.1	1.98	13.3	2.1	5.9	2.7	76.0
30	65.9	1.84	12.3	3.5	4.2	2.3	77.7
37	50.8	1.54	9.6	4.1	3.5	1.7	81.1
44	44.2	1.58	9.9	4.4	3.4	1.6	80.8
51	34.7	1.71	10.7	4.6	3.1	1.5	80.2
58	28.5	1.59	9.9	5.3	2.5	1.3	81.0
65	23.5	1.76	11.0	4.9	3.3	1.3	79.5

[a] Percent N × 6.25.

after flowering. After this period, however, moisture content decreased steadily from 88.8 to 23.5%. The protein content increased slightly from 17.6 at the 10th day to 18.6% on the 16th day, and decreased to 11.0% on the 65th day. The ether-extractable fraction and carbohydrate increased steadily with the development of the grain; crude fiber and ash content decreased up to the 37th day after flowering and remained constant afterwards. The results found here are similar to those reported by Evans (3) on United States corns.

The results of the fractionation of the total nitrogen into acid-, alkali-, and alcohol-soluble (zein) fractions, expressed on a fresh weight basis, are shown in Fig. 1. Acid-soluble nitrogen decreased up to the 30th day and increased thereafter. The decrease was from 73.3% on the 10th day to 20.4% of the total kernel nitrogen on the 30th day. Alkali-soluble nitrogen tended to increase with time. The increase was from 6.4% on the 10th day to 38.6% on the 23rd day, decreasing to 27.6% of the total nitrogen of the grain on the 44th day, with no major change thereafter. The alcohol-soluble nitrogen showed an almost linear increase with the increase in total nitrogen of the samples. The increase was from 11.8 on the 16th day to 41.5%

[3] Difco Laboratories, Detroit, Michigan.

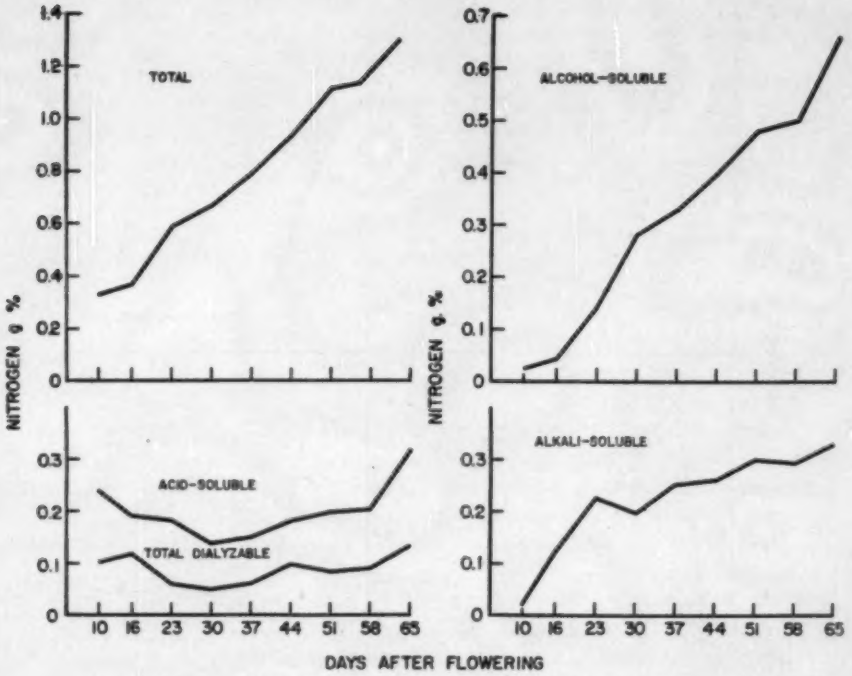

Fig. 1. Changes in nitrogen fraction composition during development of the corn kernel (fresh basis).

on the 30th day after flowering, with subsequent small variations; it reached a value of 50.6% at the end of 65 days.

In the samples collected 10 days after flowering, about 43% of the nitrogen of the acid-soluble fraction was dialyzable. The dialyzable nitrogen of the acid-soluble fraction increased from the 10th to the 16th day, decreased about half by the 23rd day, and remained nearly constant thereafter. When the dialyzable nitrogen of the acid-soluble fraction is expressed as percentage of the total nitrogen extracted from the sample, the same relationship is evident. It decreased from 31.6% on the 10th day to 10.6% on the 23rd day, and remained at about 8.0% up to the 65th day of development.

The amounts of the nitrogen fractions expressed on an absolute basis in the moisture-free samples are shown in Fig. 2. Percent total nitrogen decreased with time of development, but the acid-soluble fraction decreased sharply in about 14 days, while the alcohol-soluble nitrogen increased steadily. The alkali-soluble fraction showed a rise

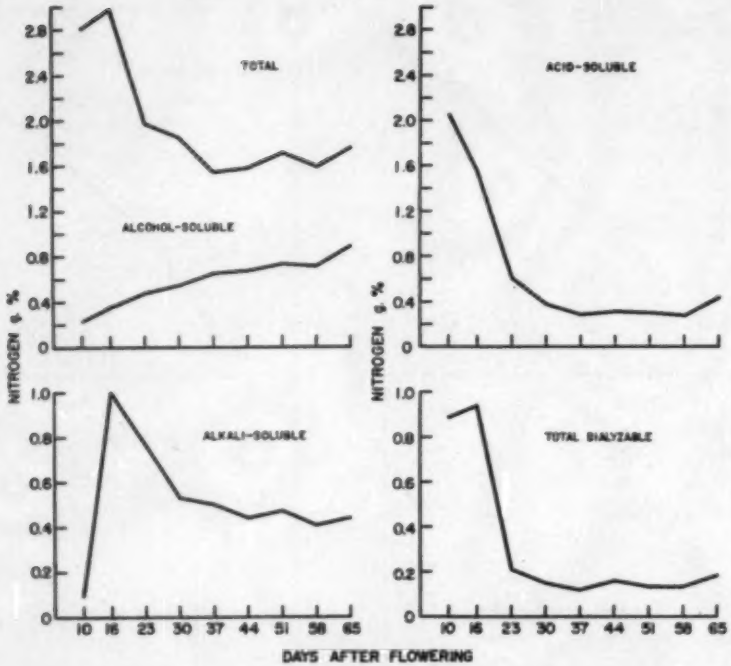

Fig. 2. Changes in nitrogen fraction composition during development of the corn kernel (dry basis).

from the 10th to the 15th day, but decreased thereafter to approximately constant values by the 30th day. Total dialyzable nitrogen varied in the same way.

The total and dialyzable nitrogen contents for the phosphate buffer extracts of the corn samples are shown in Table II. The values are averages of two determinations. The percentages of the total nitrogen extracted with phosphate buffer follow very closely the percentage of total nitrogen present as acid-soluble nitrogen fraction based on the copper sulfate fractionation. The total dialyzable nitrogen as determined from the phosphate buffer extract also follows closely the total dialyzable nitrogen of the acid-soluble fraction obtained by the copper sulfate extraction method (11,12), although the figures are slightly higher in the latter. Also shown in Table II are the values for NH_3-nitrogen and of the total free amino acids of the phosphate buffer extract. Ammonia nitrogen increased slightly from the 10th to the 16th day, from 1.05 to 1.66%, and decreased

TABLE II

PERCENTAGE OF PHOSPHATE-SOLUBLE AND OF DIALYZABLE NITROGEN, AND CONTENT OF FREE AMINO ACIDS OF THE CORN KERNEL DURING DEVELOPMENT OF THE GRAIN

DAYS AFTER FLOWERING	SOLUBLE NITROGEN IN PHOSPHATE BUFFER FROM TOTAL KERNEL NITROGEN	DIALYZABLE NITROGEN IN TOTAL KERNEL NITROGEN	NH_3 NITROGEN OF TOTAL KERNEL NITROGEN	TOTAL FREE AMINO ACIDS[a]
	%	%	%	*g/100 g*
10	60.9	30.0	1.05	3.73
16	64.7	34.8	1.66	6.61
23	35.0	15.1	1.00	1.25
30	25.0	10.7	0.59	0.47
37	21.3	8.7	0.36	0.18
44	16.1	6.4	0.25	0.18
51	12.9	4.9	0.36	0.15
58	20.0	5.5	0.21	0.15
65	19.7	4.8	0.11	0.09

[a] Equivalent to leucine and expressed on a moisture-free basis.

rapidly thereafter. The total free amino acids of the phosphate buffer extract, expressed as equivalent to leucine, increased from 0.41 g.% on the 10th day to 0.74 g.% on the 16th day, decreasing to 0.35 g.% on the 23rd day. The amount of total free amino acids decreased further, reaching an average of 0.09 g.% after the 30th day post-flowering.

Some of the amino acids of the total proteins of corn and the niacin of the grain were also determined in the different samples and the results are given in Table III. All amino acids studied increased

TABLE III

AMINO ACID CHANGES OCCURRING DURING THE DEVELOPMENT OF THE CORN KERNEL (Moisture-Free Basis)

DAYS AFTER FLOWERING	ARGININE	ISOLEUCINE	LEUCINE	LYSINE	METHIONINE	PHENYLALANINE	TYROSINE	TRYPTOPHAN	NICOTINIC ACID
	%	%	%	%	%	%	%	%	*mg %*
10	0.55	0.45	0.82	1.00	0.64	0.45	0.45	0.109	6.73
16	0.89	0.63	1.07	1.16	0.71	0.54	0.45	0.134	10.63
23	0.54	0.50	0.86	0.68	0.39	0.57	0.32	0.065	7.03
37	0.45	0.49	1.30	0.47	0.26	0.55	0.24	0.041	3.92
51	0.47	0.46	1.41	0.44	0.17	0.58	0.28	0.043	2.68
65	0.60	0.55	1.58	0.39	0.16	0.65	0.27	0.043	1.91
	g/g N	*g/g N*	*g/g N*	*g/g N*	*g/g N*	*g/g N*	*g/g N*	*mg/g N*	
10	0.19	0.16	0.26	0.32	0.21	0.16	0.14	36.4	
16	0.27	0.20	0.31	0.35	0.21	0.15	0.13	40.5	
23	0.25	0.24	0.41	0.32	0.18	0.27	0.14	30.5	
37	0.28	0.30	0.81	0.29	0.16	0.35	0.16	25.3	
51	0.28	0.27	0.82	0.26	0.10	0.34	0.16	25.0	
65	0.35	0.32	0.92	0.23	0.09	0.38	0.16	25.2	

when expressed as percentage of the dry sample, from the 10th to the 16th day. Thereafter, however, and when expressed on the basis of the nitrogen of the sample, the change in the amino acid value patterns differed. Lysine, an amino acid in which zein is notably deficient, decreased from 0.32 to 0.23 g. per g. of nitrogen from the 10th to the 65th day. Methionine also decreased. Isoleucine increased from 0.16 to 0.32 g. per g. of nitrogen from the first to the last of the samples collected. Leucine, present in large amounts in zein, increased almost fourfold from the 10th to the 65th day after flowering, paralleling the increase in zein as the kernel matures. Phenylalanine and arginine doubled in concentration during the maturation of the corn. Tyrosine, on the other hand, after a small initial increase, remained constant. The tryptophan content of the corn kernel increased slightly from the 10th to the 16th day, decreasing again to constant values beyond the 23rd day after flowering.

Because of the recognized relationship of tryptophan to niacin (14), the behavior of the vitamin during development of the grain was investigated. When the niacin is expressed on a moisture-free basis, the values rose from 6.70 to 10.58 mg. per 100 g. in 6 days, decreasing to 7.03 mg.% on the 23rd day and still further with increasing time of maturation of the grain.

No relationship between the values of niacin and tryptophan was apparent, although the maximum values for both occurred, at the same stage of maturation, on the 16th day after flowering. In general, the amino acids which are found in significant amounts in the alcohol-soluble protein of corn, zein, tend to increase with the maturation of the grain, as was the case with the alcohol-soluble nitrogen fraction, while those amino acids which are found in relatively small amounts in zein tend to decrease with time of development of the corn kernel.

Discussion

The results obtained are in agreement with those of Zeleny (21), although a different extraction and fractionation technique was used to determine the changes in the distribution of the nitrogen fraction of the corn kernel as the grain matures. Zein, the alcohol-soluble nitrogen fraction, was nearly absent in the immature kernel, becoming, however, the most important protein fraction as the grain develops and reaches maturity. This increase appears to take place at the expense of the acid-soluble nitrogen either directly or through the dialyzable nitrogen of the acid-soluble nitrogen fraction, both of which behave similarly as the grain matures. From the viewpoint of

the nutritional quality of the proteins in corn, it becomes evident that it would be difficult simultaneously to improve the quantity and quality of the protein of corn; as the nitrogen content of the grain increases, there is a parallel increase in zein which becomes the largest protein fraction of the corn kernel. Similar conclusions have been reached by other workers (4,6,8,9,19). The above statements are also supported by the changes in the concentrations of the amino acids as the grain matures, for, in general, the amino acids present in significant amounts in zein increase while those low in this fraction decrease.

Unfortunately, the amino acids present in zein in significant amounts, for example leucine, are not limiting, while those present in low concentrations, such as lysine, are limiting for animal growth in a large number of cereal grains and other food materials. Since both lysine and tryptophan are low or absent in zein (10) and both showed an initial increase from the 10th to the 16th day after flowering, the changes in concentration of these two amino acids could be related to the changes in the concentration of alkali-soluble fraction as the grain matures, since this nitrogen fraction also showed an increase on the 16th day after flowering. It has already been reported that this fraction contains more lysine and tryptophan than zein (21).

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WET-MILLING HIGH-AMYLOSE CORN CONTAINING 66- TO 68-PERCENT-AMYLOSE STARCH[1]

R. A. Anderson, C. Vojnovich, and E. L. Griffin, Jr.

ABSTRACT

High-amylose corn lots with starch containing up to 68% amylose, obtained from two corn breeders, were wet-milled on a laboratory and pilot-plant scale, to gather information pertinent to the genetic development of this new corn. Processing characteristics of an inbred sample with starch of 68% amylose content were poorer than those observed in the wet-milling of corn with lower amylose contents. Another corn sample, a hybrid with starch containing 66.7% amylose, exhibited extremely good wet-milling characteristics, contrary to a previously apparent pattern of increasingly poor processing as the amylose content of corn increased. The recovery of 82.7% of the starch present was over 10% greater than the amount recovered from corn containing 57%-amylose starch. The 0.48% protein content of the starch recovered from the 66.7%-amylose corn is comparable to that of ordinary corn starch. Differences in genetic background probably account for the marked improvement in processing characteristics of this new corn.

In a previous article we compared the wet-milling characteristics of two dent hybrid corns having starch with 49 and 57% amylose content with those of ordinary dent corn (3). Two significant differences in processing were increased swelling of the high-amylose

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corn kernel and decreased purity and yield of the high-amylose starch. These differences became more pronounced as the amylose content of the starch increased. Similar investigations were continued on several samples of high-amylose corn containing starch with 66 to 68% amylose, to provide plant breeders with information on the response of the corn samples to wet-milling and to develop optimum conditions for starch recovery. Results of these investigations are compared with those obtained in the previous work.

Material and Methods

Materials. High-amylose corn having starch with amylose contents of 66 to 68% was obtained from two corn breeders. One sample, NRRL 38225, was supplied by M. S. Zuber, Crops Research Division, Agricultural Research Service, U.S. Department of Agriculture, stationed at the Missouri Agricultural Experiment Station, Columbia, Missouri. This corn is an inbred line in early stage of development, produced from crosses giving an amylose content of about 68%.

R. P. Bear, Bear Hybrid Corn Company, Decatur, Illinois, provided the second sample, NRRL 39990, which is a field-type dent hybrid with starch containing 66.7% amylose. This corn has not yet been developed into a commercial hybrid. The history and composition of these corn samples, and of the ordinary corn used as control, are given in Table I. Although the analyses of the samples received from

TABLE I

HISTORY AND COMPOSITION OF CORN USED IN WET-MILLING EXPERIMENTS
(All percentages on a moisture-free basis)

HISTORY AND COMPOSITION	HIGH-AMYLOSE CORN (INBRED), NRRL 38225	HIGH-AMYLOSE CORN (HYBRID), NRRL 39990	ORDINARY DENT CORN (CONTROL), NRRL 18429
History:			
Plant breeder	Zuber	Bear	....
Crop year	1958	1959	1956
Composition:			
Protein, %	14.5	11.3	10.1
Crude fat, %	4.9	5.2	4.5
Starch, %	57.8	61.4	73.8
Amylose content of the starch, %	68.1	66.7	24
Total solubles, %	7.1	7.1	5.6
Total sugars, %	2.5	3.4	2.3

the two breeders are similar, the appearance of the grains is quite different. In Fig. 1, ears of the high-amylose corns are compared with an ear of the ordinary dent hybrid corn. The ear from the inbred line is considerably smaller and less colored than the hybrid ear. Kernels from the smaller ear are also small and shriveled. The

Fig. 1. From top to bottom, ear of an ordinary dent hybrid corn, NRRL 18429; ear of high-amylose dent hybrid corn (66.7% amylose), NRRL 39990; and an ear of high-amylose inbred corn (68% amylose), NRRL 38225.

hybrid ear of high-amylose corn is similar to the ear of ordinary corn.

Processing Methods. Experiments were carried out in duplicate on a laboratory scale and on a small pilot-plant scale. In the laboratory, 1,500 g. of corn were steeped in 2,800 ml. of distilled water containing 0.25% sulfur dioxide at 125°F. (52°C.). After 48 hours of steeping, the water was drained, the grain was coarsely ground in a Quaker City[2] drug mill, and the germ was recovered by simple flotation. The fibrous material remaining was finely ground in the drug mill. The resulting slurry was screened first on a 0.039-in. perforated copper sieve to remove coarse fibers and then on a 200-mesh stainless-steel sieve to remove fine fibers. Starch was recovered from the ordinary corn by a single tabling. The starch was recovered from high-amylose corn by tabling the mill starch first to remove the gluten. Then the crude starch was removed from the table, reslurried, and again tabled to recover a purer starch. This procedure for recovering starch from high-amylose corn was described previously (3).

[2]Mention of firm names or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other firms or similar products not mentioned.

In the pilot-plant experiments, 2 bu. of grain were steeped in tapwater containing sulfur dioxide. The water was drained after steeping was completed, and the corn was ground in a Bauer mill equipped with jaw-toothed plates to crack the kernel and free the germ. The germ was recovered by flotation. The remaining material was then ground in a Rietz disintegrator equipped with a 1/16-in. round, perforated screen. Coarse fibers were removed from the slurry by passing it over a Rotex gyratory shaker equipped with a 26-mesh screen, and fine fibers were recovered by passing the slurry over the same shaker equipped with a 200-mesh screen. The mill starch was separated into starch and gluten by tabling. The pilot-plant equipment and procedures have been described by Anderson (2).

Analytical Methods. The moisture of the corn and of the various recovered fractions was determined by drying a sample for 4 hours at 110°C. under a vacuum of 28 in. of mercury. Protein (N × 6.25) was determined by an improved Kjeldahl method for nitrate-free samples (1). Starch content was determined polarimetrically by the procedure of Earle and Milner (6). Apparent amylose content of the corn was determined by the iodine sorption method of Bates, French, and Rundle (5), essentially as modified by Wilson, Schoch, and Hudson (7). The amylose analysis results in figures that are generally high, as it has been shown that the amount of material which can be isolated as amylose from this type of corn is significantly lower (8). Fat analysis was carried out according to the official AOAC method of analysis (4). Solubles and sugar analyses were conducted according to AACC laboratory methods (1). Sulfur dioxide in the steepwater was determined by titration with iodine solution using starch as the indicator. All recovery and analytical data presented are on a moisture-free basis (m.f.b.).

Results and Discussion

Processing Characteristics of High-Amylose Corn (66-68%). Laboratory-scale wet-milling investigations were carried out on the two samples of high-amylose corn and on a sample of ordinary dent corn. Processing characteristics of the two high-amylose corns were compared with those of ordinary corn to show differences thought to be caused by genetic background.

Steeping characteristics were closely examined throughout the complete steeping cycle by analyzing, at intervals, the steeped corn for moisture and the steepwater for pH and sulfur dioxide content. The steeped-corn moisture-curve from both high-amylose corn samples

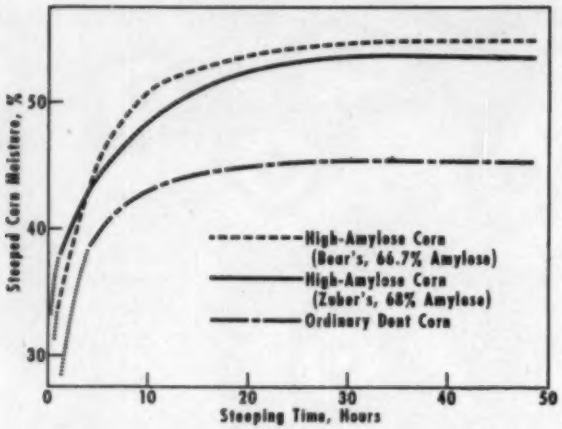

Fig. 2. Effect of steeping time on steeped corn moisture.

followed the characteristic water-absorption curves obtained from steeping ordinary corn (Fig. 2). Although the time of maximum water absorption was approximately the same for all samples, about 10 hours, the moisture content of the high-amylose corns was 7 to 9% more than that of the ordinary corn. The pH of the steepwater obtained from these corn samples followed similar curves, but the pH of the steepwater from the ordinary corn was somewhat higher than that for the hybrid sample of high-amylose corn, and was slightly lower than that for the inbred sample, as illustrated in Fig. 3. The sulfur dioxide disappearance from the steepwater was about the same for

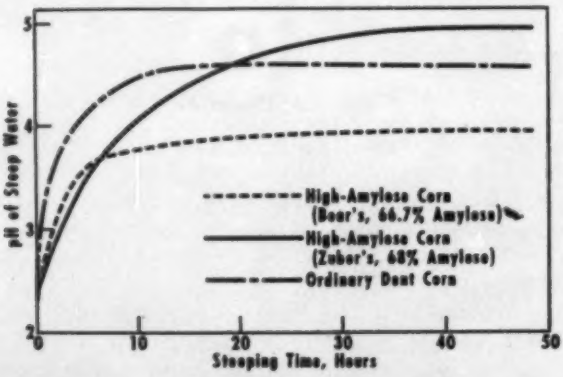

Fig. 3. Effect of steeping time on pH of the steepwater.

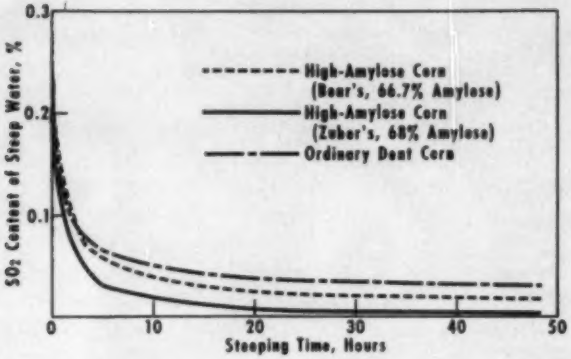

Fig. 4. Effect of steeping time on sulfur dioxide content of the steepwater.

the three samples; only a trace remained after steeping of the high-amylose corn inbred (Fig. 4). The yield of solids in the steepwater and their protein content averaged 4.1 and 30.8%, respectively, and were about the same for all three samples. The comparison of this processing characteristic and others is shown in Table II.

During steeping, the high-amylose corn inbred kernel exhibited an increase of 121% of its original dry volume; whereas the hybrid had an increase of 105%, and the ordinary corn only 63%.

The germ yield from the processing of both samples of high-amylose corn was higher than that from ordinary corn, 5.8 and 7.6%, as compared with 4.7%. The oil content of the germ from the high-amylose corn was 7 to 12% less than the 53% present in the ordinary corn germ. But total oil recovered from high-amylose corn was the same or slightly higher than that from the ordinary corn.

The response of the coarse and fine fibers to wet-milling was about the same for all three corns. However, fiber recovery from the inbred sample was considerably greater than that from the high-amylose hybrid or the ordinary corn. Because it was more difficult to wash starch from the fibers of the inbred corn, a relatively high amount of starch remained in both coarse and fine fibers. The fibers recovered from the high-amylose corn hybrid were washed free of starch quite readily, so that their starch content was less than that generally found in ordinary corn fibers.

Double-tabling the mill starch from each of the two high-amylose corns produced drastically different results. Based on starch in the inbred corn sample, the recovery of starch was only 69.7%; the starch contained 0.73% protein. The amount of starch obtained from proc-

TABLE II

COMPARISON OF THE PROCESSING CHARACTERISTICS OF THREE DIFFERENT SAMPLES OF CORN
(Average of duplicate runs)

ITEM	HIGH-AMYLOSE CORN (INBRED), NRRL 38225	HIGH-AMYLOSE CORN (HYBRID), NRRL 39990	ORDINARY DENT CORN (CONTROL), NRRL 18429
Steepwater			
Yield[a] of solids, % m.f.b[b]	3.9	4.2	4.2
Protein content, % m.f.b.	32	29	31.2
Steeped corn			
Average final moisture, %	53	55	46
Volume increase, %	121	105	63
Germ			
Yield, % m.f.b.	5.8	7.6	4.7
Crude oil content, % m.f.b.	46.2	41.2	53.0
Recovery of total oil, %	55.2	59.7	55.5
Coarse fiber			
Yield, % m.f.b.	17.9	6.7	12.8
Protein content, % m.f.b.	13.3	10.4	14.9
Starch content, % m.f.b.	20.6	9.8	19.8
Fine fiber			
Yield, % m.f.b.	7.7	7.1	6.2
Protein content, % m.f.b.	27.7	18.1	23.8
Starch content, % m.f.b.	36.2	18.9	30.6
Starch			
Yield,% m.f.b.	40.3	50.8	64.4
Recovery, % of total starch	69.7	82.7	87.3
Protein content, % m.f.b.	0.73	0.48	0.51
Gluten			
Yield, % m.f.b.	11.2	11.2	7.8
Protein content, % m.f.b.	35.5	34.6	44.2
Starch content, % m.f.b.	52.3	44.2	...
Squeegee starch[c]			
Yield, % m.f.b.	5.3	4.1	...
Protein content, % m.f.b.	14.0	9.2	...
Starch content, % m.f.b.	54.0	81.9	...
Process waters			
Yield of solids, % m.f.b.	5.7	5.4	2.5
Protein content, % m.f.b.	42.0	36.4	40.0

[a] Yield based on original weight of dry corn.
[b] Moisture-free basis.
[c] Squeegee starch is result of second tabling.

essing the hybrid sample was 82.7%; the starch contained 0.48% of protein. In neither case was a good, hard starch cake formed. As expected from analyses, the high-amylose corns yielded less starch than did the ordinary corn. Although lower yields are primarily due to the smaller amount of starch present in high-amylose corn, the presence of some small and irregularly shaped starch granules also contributes to them.

The yield of the gluten recovered from each of the high-amylose corns was 11.2%, and their protein contents averaged about 35%. However, the gluten from the high-amylose hybrid contained only 44.2% starch, as compared to a starch content of 52.3% in the high-

amylose inbred. Generally, a 7.8% yield of gluten containing 44.2% protein was obtained from the ordinary corn, which is comparable to that obtained from a commercial tabling operation. The "squeegee starch," which was the overflow or "gluten" from the second tabling of high-amylose starch, contained primarily starch.

The solids present in the process water from wet-milling the high-amylose corn amounted to 5.7 and 5.4%, or about twice the amount generally obtained from processing ordinary corn. However, these samples all had approximately the same protein content, about 40%. The larger yield of solids from the high-amylose corns can be attributed to their higher soluble content and also to the presence of very small starch granules which were not recovered in tabling operations.

Experiments carried out in the pilot plant confirmed the data, results, and observations noted in the laboratory processing of the high-amylose corns. Starch recovered in the pilot plant from the high-amylose inbred contained 0.7% protein; starch from the hybrid sample averaged 0.55% protein.

The differences in the results obtained in the processing of these two samples of high-amylose corn indicated that some changes had occurred in the raw material, since processing conditions were held constant. The wet-milling of the high-amylose inbred gave results which followed the general pattern established in the processing of corn with 49- and 57%-amylose starch; that is, as amylose content of the corn increased, starch recovery dropped and protein content of the starch increased. However, when the high-amylose hybrid was processed, an unusually good recovery of starch having low protein content was obtained. Other processing characteristics were also much improved. This hybrid sample, then, is the first high-amylose corn that has given good wet-milling characteristics.

Comparison of High-Amylose Corn Hybrid (66.7%) with Other High-Amylose Corn Hybrids Containing 49%- and 57%-Amylose Starch. Certain significant wet-milling characteristics of the high-amylose corn hybrid (66.7%) were compared with those of two high-amylose corn hybrids with starch containing 49 and 57% amylose, which were also produced by the Bear Hybrid Corn Co. (Table III). The new corn showed improvement in processing characteristics in contrast to past experiences with high-amylose corn.

The moisture content of the three steeped high-amylose corn samples ranged from 6 to 9% higher than that for steeped ordinary corn. From the pattern observed in the milling of the 49 and 57% hybrid amylose corns, the inbred corns of higher amylose content, and the

TABLE III

COMPARISON OF SIGNIFICANT WET-MILLING CHARACTERISTICS OF THREE HIGH-AMYLOSE CORN HYBRIDS AND ORDINARY CORN

	Amylose Content, Per Cent			
	24[a]	49	57	66.7
Steep corn moisture, %	46	54	52	55
Kernel volume increase, %	68	100	128	105
Recovery of total oil, %	55.5	51.4	51.0	59.7
Starch recovery, % of total starch in corn	87.3	80.0	71.4	82.7
Protein in starch, % m.f.b.	0.51	0.53	0.70	0.48

[a] Ordinary corn.

ordinary corn, it would be expected that the volume of the steeped kernels from the hybrid 66.7% amylose corn would increase, or at least remain at about 128%. However, the increase in the volume of the steeped corn kernels of high-amylose corn (66.7%) was 23% less than that of kernels from 57%-amylose corn. This smaller increase in kernel volume will benefit corn processors by permitting a larger quantity of high-amylose corn (66.7%) to be steeped in their present equipment.

The recovery of total oil was significantly greater from the 66.7%-amylose corn than from any of the other corns, including the ordinary dent sample. Almost 60% of the oil was recovered in the germ fraction, as compared to 51% from the other high-amylose corns and 55% from ordinary corn. It is recognized that these recoveries are somewhat lower than those generally obtained by commercial processing, but the comparisons show an improved germ oil recovery from the corn containing 66.7%-amylose starch.

In previous studies the starch-gluten separation always became more difficult as the amylose content of the corn increased. Processing of high-amylose corns (49 and 57%) showed a recovery of starch of 80.0 and 71.4%, respectively. The protein content of the starch from the 49%-amylose corn was 0.53%, and from the 57% material, 0.7%. The 82.7% recovery of starch from 66.7%-amylose corn is over 10% higher than experienced in the processing of 57%-amylose corn, and comes within 4.5% of our optimum starch recovery obtained from the milling of ordinary corn. Also, the protein content of the starch recovered from high-amylose corn (66.7%) was about the same as in ordinary corn starch.

While the over-all processing results obtained with 66.7%-amylose corn were not quite as good as those obtained with ordinary dent corn, processing quality was reasonably good and improvements shown over the milling of 49%-, 57%-, and other high-amylose corns were very significant. A survey of the genetic background of

the 66.7%-amylose corn revealed that its "pedigree" had been altered to introduce more favorable characteristics to the corn[3]. This genetic control of milling quality of high-amylose corn, concurrent with increased amylose content, shows promise for future development of high-amylose corns with excellent processing characteristics, similar to those of ordinary corn.

Acknowledgments

The authors are grateful to J. G. Pemberton for his assistance in carrying out the experimental work; to D. E. Uhl, A. Clark, and L. T. Black for analyzing the many samples; to W. L. Deatherage for the amylose analysis of the corn; and to R. P. Bear and M. S. Zuber for supplying the high-amylose corn samples.

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A NOTE ON THE EFFECT OF STEEPING TIME ON WET-MILLING HIGH-AMYLOSE CORN CONTAINING 57-PERCENT-AMYLOSE STARCH[1]

R. A. Anderson, C. Vojnovich, and E. L. Griffin, Jr.

A recent paper by the authors reported that high-amylose corn exhibited a considerably greater volume increase during steeping than did ordinary corn (2). Kernels of high-amylose corn with starch containing 57% amylose increased 128% over their original dry volume, as compared with a 63% increase in volume during steeping of ordinary corn. In a commercial run on the same high-amylose corn, it was observed that swelling took place in a relatively short period of time. It was thought that steeping might be completed at this time, i.e., when the high-amylose corn kernels swelled to the same degree as the ordinary corn. This consideration prompted a short study on a laboratory scale to determine the effect of steeping time on the processing of high-amylose corn with starch containing 57% amylose.

Fifteen hundred-gram batches of corn were steeped in distilled water containing 0.25% sulfur dioxide at 125°F. (52°C.) for the required period of time. At the completion of steeping, the water was drained and the grain was ground in a Quaker City[2] drug mill. Germs were recovered by flotation, and the remaining fibrous material was again ground in the drug mill. To remove coarse fibers, the resulting slurry was screened on a 0.039-in. perforated copper sieve. It was then rescreened on a 200-mesh stainless-steel sieve to remove fine fibers. The germ and fiber fractions were washed to remove starch. The final slurry, the mill starch, was tabled twice to separate the starch from the gluten.

Protein (N × 6.25) in the various fractions was determined by an improved Kjeldahl method for nitrate-free samples (1). Starch content was determined polarimetrically by the procedure of Earle and Milner (4). Fat analysis was carried out according to the official method of analysis of the AOAC (3). Moisture was determined by drying the samples for 4 hours at 110°C. under a vacuum of 28 in. of mercury.

The chemical composition (on a moisture-free basis) of the corn used was:

[1] Manuscript received February 5, 1960. Contribution from the Northern Regional Research Laboratory, Peoria, Illinois. This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

[2] Mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

	%
Protein	13.9
Crude fat	7.1
Starch	60.9
Solubles	8.6

TABLE I

RECOVERY AND ANALYTICAL DATA FROM THE WET-MILLING OF HIGH-AMYLOSE CORN

FRACTION AND ANALYSIS MADE	STEEPING TIME, HOURS			
	16	24	36	48
Steepwater				
Yield[a] of solids, % MFB[b]	2.3	2.8	3.2	3.6
Protein content, % MFB	22.8	26.8	27.6	31.0
Steeped corn				
Average final moisture, %	51	51	51	52
Volume increase, %	126	124	131	128
Germ				
Yield, % MFB	7.6	7.3	7.6	6.5
Crude fat content, % MFB	49.4	48.7	46.3	56.0
Recovery of total oil, %	51.0	50.0	49.8	51.0
Coarse fiber				
Yield, % MFB	12.1	9.3	9.8	12.6
Protein content, % MFB	11.8	11.1	10.9	10.4
Starch content, % MFB	14.9	11.2	12.9	12.4
Fine fiber				
Yield, % MFB	5.9	7.0	7.7	4.9
Protein content, % MFB	23.7	21.2	18.9	21.1
Starch content, % MFB	29.1	24.5	25.6	30.1
Starch				
Yield, % MFB	41.0	41.5	42.0	43.5
Recovery, % of total starch	67.3	68.2	69.0	71.4
Protein content, % MFB	0.7	0.7	0.8	0.7
Gluten				
Yield, % MFB	16.7	19.1	16.6	15.5
Protein content, % MFB	33.0	30.6	25.6	33.1
Starch content, % MFB	56.4	55.6	56.0	52.3
Squeegee starch[c]				
Yield, % MFB	5.4	3.3	6.3	4.9
Protein content, % MFB	12.3	7.9	15.6	5.0
Starch content, % MFB	82.6	88.2	71.5	94.4
Process waters				
Yield of solids, % MFB	7.1	6.7	6.5	6.9
Protein content, % MFB	34.6	36.3	30.0	44.5

[a] Yield based on original weight of dry corn.
[b] Moisture-free basis.
[c] Squeegee starch is result of second tabling.

The corn was steeped for 16, 24, 36, and 48 hours and then processed under the described conditions. The recovery and analytical data are given in Table I.

Several differences were noted during the processing of the high-amylose corn steeped for the four time periods. The amount of solubles removed from the corn in 16 hours was about 36% less than that removed during the full steeping period of 48 hours; there was a

2.3% yield of solids at 16 hours, as compared to 3.6% at 48 hours. Similarly, the protein content of the steepwater solids was considerably less when the corn was steeped only 16 hours; 22.8% compared to 31% when the steeping time was 48 hours.

Water absorption by the corn during steeping was the same for all tests, and increase in volume of the corn after steeping was also the same for the four different steeping times.

Germ separation was slightly better when the corn was steeped for 48 hours. The recovered germ was freer of fiber and had a higher oil content, 56%, as compared to about 49% for the 16-hour steep. This increase is probably due to the more complete removal of solubles in the 48-hour steeping. The recovery of total oil was about the same, but less germ was needed to yield the oil when the 48-hour steep was used. No particular difficulties were encountered during separation of fibers from the mill starch.

The yield of starch from corn that was steeped for 48 hours was materially higher than that from corn steeped for shorter periods; i.e., 43.5%, as compared to 41.0, 41.5, and 42.0%, respectively, for 16, 24, and 36 hours. With the protein content of the recovered starch about the same in all experiments, this increase would be important to the wet-miller. There were no significant differences observed in processing gluten, squeegee starch, and process-water fractions. Of note is the protein content of the process-water solids, which was about 10% higher for the 48-hour run than it was in the 16-hour test.

This study indicates that this high-amylose corn is not completely steeped after 16 hours, even though it has absorbed its optimum moisture and has experienced a considerable increase in volume. A 48-hour steeping period is required to obtain complete removal of solubles and a maximum yield of starch. However, shorter steeping could be used in certain cases where, for economic reasons, the small loss in solubles and starch could be taken in lieu of increasing steeping capacity of existing facilities. Also, the utilization of countercurrent steeping as carried out by the corn refining industry should permit a shorter steeping time, but this operation should be shown on an industrial scale when sufficient high-amylose corn is available.

Acknowledgments

The authors are grateful to J. G. Pemberton and L. H. Burbridge for their assistance in the experimental work. The 57% high-amylose corn was provided by the American Maize-Products Co., Roby, Indiana, and the National Starch and Chemical Corp., Plainfield, N. J. The corn was grown from strains developed by Bear Hybrid Corn Co., Inc., Decatur, Ill.

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LABORATORY MILLING
I. A Small-Scale Bran Finisher[1]

H. C. Black, M. H. Fisher, and G. N. Irvine

ABSTRACT

A small bran finisher is described for reclaiming from bran the 1 to 3% of flour normally lost in laboratory milling. Total flour may thus be increased to approximate the yield obtained in commercial milling.

In assessing the quality of wheat samples it is often useful to obtain a yield of flour approximating that obtained in commercial practice. One reason for the lower yield usually obtained in laboratory milling is that no provision is made for reclaiming flour from the bran and shorts, whereas bran finishers and shorts dusters are normal components of commercial milling equipment.

A small-scale bran finisher has recently been built in this laboratory. It is described in this paper, and examples of results obtained with it are presented.

General Description

Figure 1 shows a photograph of the equipment. Exterior dimensions of the finisher are 32¼ by 24 by 10 in., and its weight is 143 lb. The housing is constructed from ¼-in. 24 S.T. aluminum panels, butted together and joined with ½-in. self-tapping screws. These panels were given an engine finish on the outside surfaces. A Plexi-

[1] Manuscript received August 1, 1960. Paper No. 189 of the Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg 2, Canada.

glas door, hinged to the cabinet, permits the operator to view stock passing through the screen, and gives access for cleaning. The complete unit is mounted on a 24-in. welded angle-iron base equipped with four casters. It is so constructed that it may be readily dismantled and reassembled should repairs or modifications be necessary.

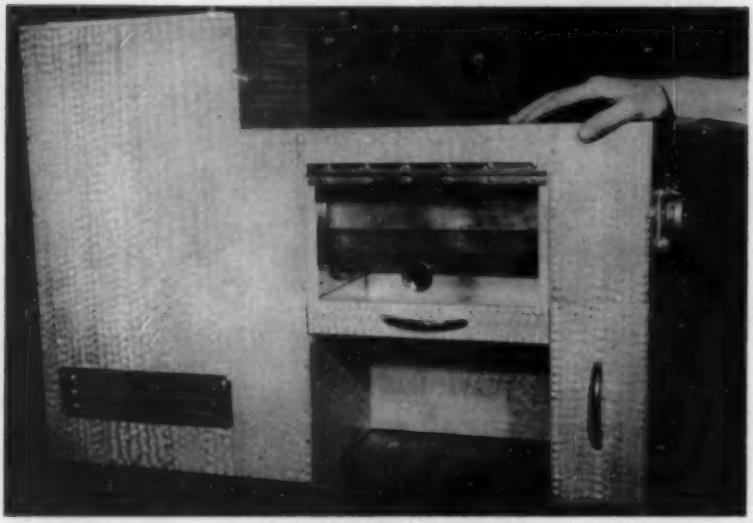

Fig. 1. Photograph of bran finisher.

Description of Principal Parts

Figure 2 is a drawing of the exposed front and end views of the machine. The taller section, on the left in the front view, houses: a feed hopper, *A*, 6½ in. deep and tapered from 12 by 9½ in. to 6¾ by ¾ in.; a ¾ in. feed worm, *B*; a variable pitch pulley, *C*, mounted on the main shaft; and a double-shafted ⅓-h.p. motor, *D*, which drives both the feed worm and the main shaft. The remaining section contains a prefeeding chamber, *E*, of circular cross-section 6 in. in diameter and 3 in. long, and an extraction chamber, *F*, 12 in. long. A four-bladed reel, *G*, 15 in. long, rotates within the prefeeding and extraction chambers. The rotor blades are 15 in. long, 1½ in. wide, and ⅛ in. thick. The outer edge of each blade is cut into teeth 1 in. long, and each tooth is divided by a ⅛-in. slot ½ in. deep. Each tooth is offset 10 degrees, which produces a spiral centrifugal action to the stock and carries it through the machine. Set

screws, *H*, permit the diameter of the reel to be adjusted up to 6 in. The reel is mounted on a ¾-in. shaft, *I*, in two self-aligning flange ball-bearing units, and is driven at 1500 r.p.m. The feed worm is driven at 425 r.p.m.

Extracted flour is collected in a drawer, *J*, below the extraction chamber, and the bran continues to the tail end of the machine,

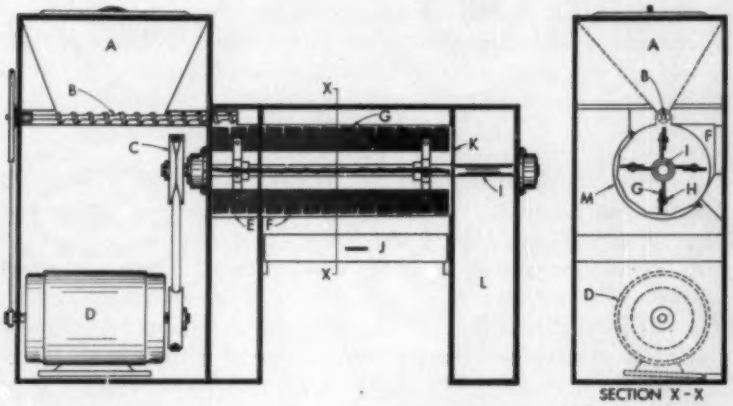

Fig. 2. Drawings of front and end views with plates removed to show interior detail.

where it is collected in another drawer, *L*. A baffle, *K*, is placed on the end of the extraction chamber to control the bran as it passes into the drawer.

The end view, on the right, shows additional details of the extraction chamber and the reel. The extraction chamber is a continuation of the circular prefeeding chamber, but is extended and offset 90°, making a flat wall on the back of the chamber. This breaks the motion of the stock and prevents it from traveling at the same speed as the rotor. As the stock is rotated and thrown against this wall, the impact aids in removing some of the adhering flour; in this respect, the laboratory machine is similar to the Buhler Impact Finisher. The front portion of the extraction chamber comprises a perforated metal screen, *M*, with holes 1 mm. in diameter, mounted on a removable frame. The surface of this screen is divided into six flats, which improves its efficiency in removing flour from the bran. The screen may be replaced with others of different mesh should this be required. Also shown in the drawing are the shaft, *I*, adjusting screws, *H*, and motor, *D*, all of which were previously described.

Operation

Bran is introduced into the feed hopper, *A*, and is carried by the feed worm, *B*, into the prefeeding chamber, *E*, where it gains momentum before entering the extraction chamber, *F*. From the extraction chamber, flour is expelled into the drawer, *J*, and bran continues through the machine and is collected in the drawer, *L*.

The bran is usually passed twice through the machine. A sample of 350 g. of bran, obtained by milling 2,000 g. of wheat, passes through the machine in approximately 1.5 minutes. There is little damage to the bran, so that contamination of the flour by bran powder is not excessive.

Typical Results

To illustrate the effectiveness of the equipment in reclaiming additional flour, examples representing two hard and two soft wheats are reported in Table I. Each wheat was a composite of its respective class. They were milled to yield on an Allis-Chalmers laboratory mill (three sets of corrugated and one set of frosted rolls), and the bran put through the finisher. Experience with a large number and wide range of samples shows that the amount of flour recovered from hard wheats is of the order of 1.2 to 2.0%, whereas about 3% or more is generally recovered from the bran of soft wheats. It should be mentioned that the mill is adjusted for hard wheats and tends to give lower yields from soft wheats than could be obtained by suitable readjustment of the mill.

Data in Table II show typical results for laboratory milling of No. 2 Manitoba Northern. A number of replicate millings were made with the Allis-Chalmers mill in order to accumulate sufficient bran-finisher flour for analytical and baking tests. The flour obtained from the mill, from the bran finisher, and from the two combined in accordance with the extraction rates, were analyzed, and were baked

TABLE I

EXAMPLES SHOWING FLOUR EXTRACTION, ASH, AND COLOR, FROM DIFFERENT CLASSES OF WHEAT

PROPERTY	HARD RED SPRING	HARD RED WINTER	RED WINTER	SOFT WHITE SPRING
Bushel weight, lb.	64.0	64.5	62.0	64.8
1000-kernel weight, g.	32.4	28.1	29.8	36.4
Mill extraction, %	72.9	72.0	67.9	68.5
Finisher extraction, %	1.5	1.7	3.2	3.2
Total extraction, %	74.4	73.7	71.1	71.7
Ash, %	0.48	0.51	0.41	0.40
Color, units	1.8	3.5	2.7	1.1

by the Remix procedure (1). The bran-finisher flour shows the high protein content, high ash, poor color, and moderate baking strength expected of this class of flour. Since the amount of bran-finisher flour, in relation to the yield of flour from the mill, is small, there is little change in flour quality when the former is added to the latter.

TABLE II

QUALITIES OF FLOUR FROM FINISHER, COMPARED WITH FLOUR FROM MILL AND TOTAL FLOUR

PROPERTY	FLOUR FROM FINISHER	FLOUR FROM MILL	TOTAL FLOUR
Extraction, %	1.5	72.5	74.0
Protein, %	20.4	12.0	12.1
Wet gluten, %	63.9	35.8	36.1
Ash, %	1.63	0.48	0.50
Color, units	11.8	1.5	1.9
Diastatic activity, mg.	197	229	224
Gassing power, mm.	480	430	435
Baking absorption, %	61.7	60.1	60.2
Loaf volume, cc.	695	810	790
Appearance	2.0	9.7	10.0
Crumb texture	1.3–co	6.2–o	6.3–o
Crumb color	Brown	8.0	7.8

The bran finisher described in this paper has been in use in this laboratory for almost two years. Both operation and maintenance are simple. It has been used more especially for assessing the quality of commercial shipments of wheats of different grades. In this application, the advantage of using extraction rates approximating those obtained commercially are readily apparent. The bran finisher has also been used for investigating the milling properties of varieties. Since the machine may prove useful in other laboratories, a description of it seems warranted.

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EDITORIAL POLICY

Cereal Chemistry publishes scientific papers dealing with raw materials, processes, or products of the cereal industries, or with analytical procedures, technological tests, of fundamental research, related thereto. Papers must be based on original investigations, not previously described elsewhere, which make a definite contribution to existing knowledge.

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Manuscripts for publication, and correspondence directly pertaining to them, should be sent to the Editor-in-Chief, CEREAL CHEMISTRY, 1955 University Ave., St. Paul 4, Minn. All other correspondence, including circulation, advertising, or other business, should be directed to the Managing Editor at the above address.

Manuscripts of published papers will be kept on file for one year. After that time they will be destroyed unless other instructions have been received from the author. Original graphs, etc., and negatives of all illustrations are returned to the author immediately upon publication.

SUGGESTIONS TO AUTHORS

General. Authors will find the last volume of *Cereal Chemistry* a useful guide to acceptable arrangement and styling of papers. "On Writing Scientific Papers for Cereal Chemistry" (*Trans. Am. Assoc. Cereal Chemists* 6:1–22. 1948) amplifies the following notes.

Authors should submit two copies of the manuscript, typed double spaced with wide margins on 8½ by 11 inch white paper, and all original drawings or photographs for figures. If possible, one set of photographs of figures should also be submitted. Originals can then be held to prevent damage, and the photographs can be sent to reviewers.

Editorial Style. AACC publications are edited in accordance with *A Manual of Style*, University of Chicago Press, and *Webster's Dictionary*. A few points which authors often treat wrongly are listed below:

Use names, not formulas, for text references to chemical compounds. Use plural verbs with quantities (6.9 g. were). Figures are used before abbreviations (3 ml.), and % rather than "per cent" is used following figures. All units are abbreviated and followed by periods, except units of time, which are spelled out. Repeat the degree sign (5°–10°C.). Place 0 before the decimal point for correlation coefficients ($r = 0.95$). Use * to mark statistics that exceed the 5% level and ** for those that exceed the 1% level; footnotes explaining this convention are no longer required. Type fractions on one line if possible, e.g., $A/(B + C)$. Use lower case for farinograph, mixogram, etc., unless used with a proper name, i.e., Brabender Farinograph. The term *extensigraph* is to be used exclusively in papers which present experimental data obtained with either the Brabender Extensograph* (manufactured by the Brabender Corporation, Rochelle Park, N.J.,) or the Brabender Extensigraph (manufactured by C. W. Brabender Instruments, Inc., South Hackensack, N.J.). When in doubt about a point that occurs frequently, consult the Style Manual or the Dictionary.

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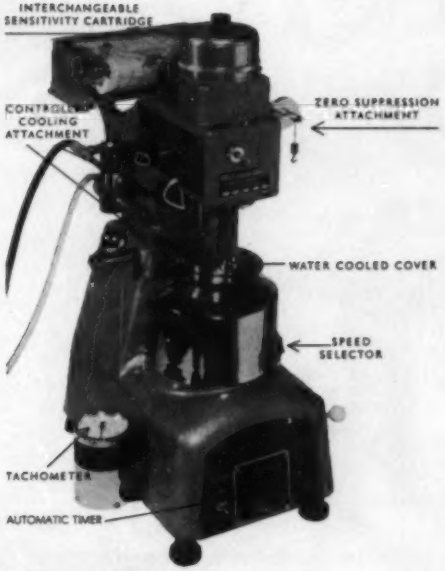

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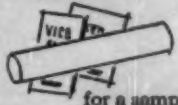

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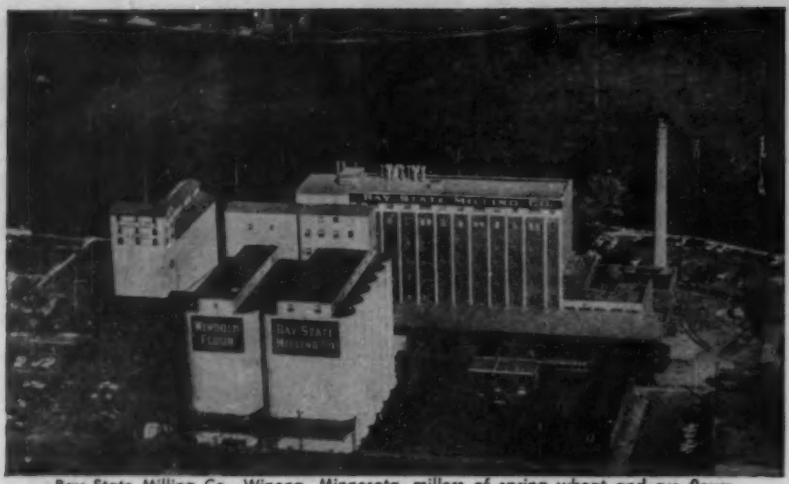

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